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16. ABSTRACT

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A list of compounds, representative of potential contaminants, was used to evaluate commercial, military and specially designed procedures which have application to water testing. Following the original screening, promising methods were further evaluated against samples of natural waters polluted in the laboratory with compounds from the model list. It was concluded that a selection of 15 multiple non-specific detection systems could be organized into a detection concept which would detect a significant portion of potential contaminants. A "paper analysis" projected that about 85% of potential contaminants would respond to at least one detection parameter.

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FOREWORD

When energy and material resources are extracted, processed, converted, and used, the related pollutional impacts on our environment and even on our health often require that new and increasingly more efficient pollution control methods be used. The Industrial Environmental Research Laboratory - Cincinnati (IERL-Ci) assists in developing and demonstrating new and improved methodologies that will meet these needs both efficiently and economically.

This report is a product of the above efforts. It documents the laboratory and field studies conducted in the development of a field kit for detecting spills of hazardous materials in watercourses. The kit, consisting of 15 different chemical tests packaged in a one-man portable configuration is capable of detecting a wide variety of polluting substances in water.

This report should be of value to Federal, state and local government personnel as well as to individuals from the chemical process and transportation industries who are involved in responding to accidental releases of hazardous substances. Information on this subject beyond that supplied in the report may be obtained from the Oil and Hazardous Materials Spills Branch (IERL), Edison, New Jersey 08817.

David G. Stephan
Director
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ABSTRACT

Chemical Systems Laboratory, under a program sponsored by the Environmental Protection Agency, Edison, NJ has developed a kit to detect hazardous material spills into waterways. The purpose of the program was to develop a man-portable field kit able to detect (not necessarily identify) as many contaminants in water in as low a concentration as possible.

A list of compounds, representative of potential contaminants, was used to evaluate commercial, military and specially designed procedures which have application to water testing. Following the original screening, promising methods were further evaluated against samples of natural waters polluted in the laboratory with compounds from the model list. It was concluded that a selection of 15 multiple non-specific detection systems could be organized into a detection concept which would detect a significant portion of potential contaminants. A "paper analysis" projected that about 85% of potential contaminants would respond to at least one detection parameter.

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Personnel of Chemical Detection and Alarms Branch who typed the report.

SECTION 1
INTRODUCTION

Chemical Systems Laboratory, under the sponsorship of the Environmental Protection Agency (EPA), Oil and Hazardous Materials Spills Branch, Industrial Environmental Research Lab, Edison, NJ, developed a field kit to detect (not necessarily identify) hazardous material spills into inland waterways. Development was implemented by surveying reported procedures, making original studies and evaluating Army and industrial equipment which could be used for detection of contaminants in water. Using a model list of contaminants, procedures were then selected to give a total detection concept. Finally, necessary instruments and equipment to adapt the procedures to field use were chosen and designed into a man-portable kit. Two prototype kits were fabricated for EPA complete with drawings, manuals and parts lists.

BACKGROUND

Every year in the United States there are over 3,000 spills of hazardous polluting materials (other than oil) into waterways. These include in-plant and storage spills, as well as spills caused during transportation of materials by barge, tank truck, railway tank car and pipeline. In addition, agricultural use of chemicals and run-off of natural waters deposit large amounts of materials into rivers, streams and lakes. The materials, many of which of which are hazardous to public health and wildlife, include alcohols, pesticides, phosphates, nitrates, sulfates and a variety of other industrial organic and inorganic chemicals. With the number of incidents increasing every year, an urgent need exists for a simple man-portable field kit capable of detecting contaminants in waterways.

At the present time EPA is concerned with detection of about 370 hazardous materials published in the Federal Register, Vol. 40, No. 250 of 30 December 1975.

A number of specific points were considered during development:

1. In most cases the nature of the spill will be known; it will be necessary to detect its "plume" down the waterway until countermeasures can be taken or the contamination disappears. Non-specific detection methods which are generally responsive to multiple contaminants and can be related to concentration are needed. High sensitivity (while desirable) is not always the most important criteria in tracing a "plume". Lengthy procedures are to be avoided since a large number of repetitive tests at short intervals will be required.

2. The kit will frequently be used from a small boat rather than shore. Therefore, techniques requiring complex equipment or open heat are unsuitable.

3. Methods requiring little or no makeup reagents are preferred, to reduce the size of the kit and the logistical burden.

4. EPA prefers shelf-available instrumentation, equipment and prepackaged reagents, since they have a limited staff and propose to maintain the kits themselves.

APPROACH

It was proposed to develop a total detection concept using a selection of non-specific tests with a broad detection response for as many contaminants as possible.

It was decided to make the kit as versatile as possible so that it could be modified or added to as requirements change.

The large number of materials of concern to EPA made it necessary to develop a model list of contaminants for evaluation studies. It was agreed to use findings from this evaluation for a "paper analysis" (Appendix A) to indicate the probability of detection of other materials from the Federal Register list.

A first estimate of detection capabilities for the field was made from standard references[1,2,3,4]. Literature was obtained from water test kit suppliers and a review was made of Army equipment and devices which could be applied to detection of contamination in water. It was immediately evident that tests for inorganic components are well established, and in many cases prepackaged reagent systems are available. Tests for organic materials are comparatively few, and often are not simple nor do they lend themselves to repetitive use. This area required the most new development work.

To reduce the need for refill and special reagents, enphasis was placed on instrumental methods. These included pH meters, conductivity meters, ultraviolet light methods, colorimeters and ion-selective electrodes. This approach requires a power supply, but the advantages easily outweigh the inconvenience.

In reviewing methods or instruments to be used in the kit, a comprehensive survey of all available selections was not made. When possible, several choices were compared, then the most reliable and/or one most easily worked into a detection concept was selected. Thus, on finding a reliable pH meter, phosphate test or detector tube, that search ended and other detection requirements were addressed.

Because of the low solubility of many of the materials, concentration techniques were also studied.

Detection of contaminants in natural waters is complicated by the fact many interfering ions and materials are present as background components. If the contaminant is a natural component of water (e.g. sulfate ion) it is necessary to detect its presence by a gain over background. This can be done by comparing the contaminated water with a reference sample. A reference sample is taken at a point upstream from the contaminated area, or in the case of a lake or a pond, at an area removed from the spill area. Once a reference is established, the "plume" is traced by the change in water quality.

The program used to develop a detector kit for hazardous material spills is outlined below:

PROGRAM

PHASE I.
- A. Select model list of contaminants.
- B. Evaluate commercial, military and specially designed tests.

PHASE II.
- A. Evaluate selected methods against contaminants in natural waters.
- B. Survey background level of natural waters with candidate methods.
- C. Select methods and formulate concept.
- D. Prepare "paper analysis" of Federal Register listing.

PHASE III.
- A. Fabricate kits, draft manual.
- B. Conduct fieldability tests.
- C. Finalize manual, prepare drawings and parts lists.
- D. Prepare final report.

SECTION 2
CONCLUSIONS

Chemical Systems Laboratory personnel have developed a field kit for detection of hazardous material spills into inland waters. Prototypes have been prepared and provided to EPA along with manuals, parts lists and drawings. While the kit is not a panacea for all spill situations, in the hands of a dedicated investigator its instrumentation and chemistry can provide a variety of valuable data which will help him accomplish many of his tasks.

The makeup of the kit is simple enough so modifications may be made to suit particular needs and applications. In fact, as information on its performance is received from the field, changes may be made to the detection concept itself.

This program is a start in assessing the overall nature of contamination and the problems associated with its detection. Areas of detection weaknesses and suggested leads for continued investigations are noted in Section 3.

SECTION 3
RECOMMENDATIONS

The hazardous materials detector kit concept has excellent potential for detection of contamination in inland waters. The kit is versatile and easily modified for special applications.

It is apparent that a number of gaps exist in the detection systems, particularly for many organic compounds. This further reflects the limited capability of commercial kits in dealing with organic contamination in water. The detection gaps found in the "paper analysis" reflect the same shortcomings.

Certain areas showing promise but not brought to conclusion within the time frame of this program, are felt to be worthy of continued study.

a) Methods using open cell polyurethane cubes impregnated with chromogenic reagents and prepackaged in plastic tubes are promising. One of the processes developed, used urethane cubes loaded with dithizone to detect heavy metals. Stability problems which were not resolved prevented their inclusion in the detector kit. Use of anti-oxidants such as ascorbic acid may be of value in stabilizing them. Similar devices employing the 4-aminoantipyrene reagent for phenol were prepared. However, time was not available at the end of the program to determine their stability.

b) Compounds known as fluorescent probes are promising detectors for high molecular weight organic materials. This system would be especially useful because it is unrelated to other detection methods and would add a new dimension to the detector kit. The problem to be resolved is one of developing simple universal extraction and concentration techniques.

c) Methods using silver salts to detect organochlorine materials would be useful since a significant number of these compounds are listed as hazards in the Federal Register. The problem again, is with extraction and concentration techniques.

d) It is evident from the above that continued studies in the area of extraction and concentration techniques would be of general overall application to detection of contaminants in water.

e) Enzyme systems, similar to that included in the kit may be designed to more specifically respond to insecticides and other industrial contaminants.

f) Certain detector tubes were not considered for use in the effervescent process developed for the detector kit because they do not have multiple response capability; this doesn't preclude their evaluation and use for specific contaminants.

Finally, the kit is simple and versatile in makeup so that modifications may easily be made. In fact, Anderson Laboratories Corporation, Fort Worth, TX, carries all the reagents necessary to perform tests in the 13th Edition of Standard Methods for the Examination of Water, many of which are potentially adaptable to the kit.

SECTION 4
EXPERIMENTAL STUDIES

MODEL LIST OF CONTAMINANTS

As indicated, the large number of materials of concern to EPA made it necessary to develop a model list of contaminants for evaluation studies. Included were the most important materials from a hazards standpoint[5] and others representative of the complete list. The materials selected are shown in Table 1.

Solutions for evaluation of candidate detection methods were prepared by mixing a one gram sample of the contaminant in one liter of water. When the contaminant is totally soluble, the concentration of the initial solution is 1000 mg/l; for highly insoluble compounds, the concentration of the initial solution is assumed to be that of a saturated solution as reported in the literature. Three additional dilutions of 1:10, 1:100 and 1:1000 were made from the clear supernatant liquid and the concentrations were calculated from the saturation values.

Candidate tests were first screened against the highest concentration of contaminant. When a positive response was noted, lower concentrations were further tested until a negative response was obtained or all four concentrations were tested. Detection tests were applied to all solutions, regardless of whether a response was expected. In this manner, many cross-interferences were observed which were useful in a multiple response capability concept.

EVALUATION OF METHODS

Methods are grouped by the process used or the chemical or physical measurement made. Sections are cross-referenced where approriate. Preparations and testing of the contaminant solutions have been described in Section 4.

Reasons for inclusion or rejection of individual tests are discussed. Response of the tests selected for the kit, to the model list of contaminants is shown in Table 2. Information on the selected methods is summarized in Section 5. Detailed procedures for each test are given in the Operator's Manual prepared for the kit (Appendix B).

Table 1

Model List of Hazardous Materials

(Concentration of Initial Solution)

	Hazardous Substance	mg/l		Hazardous Substance	mg/l
1.	Phenol	1000	18.	Toxaphene	3
2.	Methanol	1000	19.	DDT	0.005
3.	Acrylonitrile	1000	20.	EPN	500
4.	Chlorosulfonic Acid	1000	21.	Malathion	145
5.	Benzene	1000	22.	Parathion	25
6.	Ammonium Chloride	1000	23.	Dieldrin	0.25
7.	Phosphorus Pentasulfide	1000	24.	Heptachlor	0.2
8.	Styrene	660	25.	Sevin	40
9.	Acetone Cyanohydrin	1000	26.	Chlordane	0.1
10.	Calcium Hypochlorite	1000	27.	Fermate	120
11.	Nonylphenol	17	28.	Lead Arsenate	2.5
12.	Isoprene	100	29.	Disodium Methyl Arsenate	1000
13.	Xylenes	130	30.	Phenyl Mercuric Chloride	6.9
14.	Nitrophenol	1000	31.	2,4-D (Acid)	900
15.	Ammonium Nitrate	1000	32.	2,4,5-T (Acid)	280
16.	Aluminum Sulfate	1000	33.	Ammonium Phosphate,Dibasic	1000
17.	Aldrin	0.2			

Certain detection approaches recur in this evaluation. For example, quantitative measurements were made using color comparators, the Hach Chemical Company DR Colorimeter and the Hach DR-EL/2 Spectrophotometer. Color comparators require visual matching of developed colors with a series of permanent glass or plastic standards. The process is highly subjective and under some conditions may only be semiquantitative. Since accurate quantitative measurement is necessary for many situations, an instrumental method is preferred.

The Hach DR Colorimeter is a portable battery-operated filter colorimeter. The Hach Spectrophotometer, which is also portable and battery operated (Appendix B, p. 9) has a simple variable filter calibrated in nanometers which precludes the need to change filters for different analyses.

Hach liquid reagents are packaged with calibrated dropping pipettes. Dry reagents are packaged with automatic measuring devices or in individually sealed plastic tubes which are opened with clips to obtain the reagent.

The tests evaluated in this investigation are summarized below:

a. Acidity. Acidity was determined in the conventional manner by titration with 0.02N NaOH to a phenolphthalein endpoint[3]. The "drop-count" titration method was used to simplify handling for the field. It was found that acidity data parallels data obtained with a pH meter. A pH meter is preferred for simplicity of use, reliability of data and use of fewer chemicals; the acidity test was rejected.

b. Alcohol. Chemical tests for methyl alcohol in water based on cerric ammonium nitrate[6] and vanadium oxinate[7] were investigated but did not respond at the 1000 mg/l level. Other alcohol tests based on enzymatic methods are described under Enzyme Systems (Section m). Several tests using detector tubes are discussed under Detector Tubes (Section 1). An alcohol test as such was not used.

c. Alkalinity. Alkalinity was measured by titration with 0.02N H_2SO_4 to a mixed bromcresol green-methyl red endpoint[3] using the "drop-count" titration method. Reasons for its rejection are the same as for the Acidity test (Section a).

d. Ammonia Nitrogen. The Ammonia Nitrogen Tester, Model NI-8, marketed by the Hach Chemical Company was evaluated. The kit uses the Nessler reagent to develop color which is measured by comparison to colored standards. Use of comparators for the kit was considered too subjective for quantitative analysis, particularly under variable light conditions. The same procedure using either a colorimeter or spectrophotometer is preferred. See data Table 2 and procedure (Appendix B, p. 21). The test responds to a wide range of organic and inorganic compounds.

Other compounds, which responded to the Nessler reagent at 100 mg/l or less are chloramine-T, dichloroamine-T, dicyclohexylamine, 2,4-dinitrophenylamine, 2-ethylhexylamine, hexamethylenimine, N-methylglucamine, 2-nitrodiphenylamine, uric acid and m-xylenediamine.

A positive response with the Nessler reagent is indicated by formation of a yellow color or a precipitate.

e. Chloride. Commercial tests for chloride lack sensitivity or require titration making them unsuitable for the kit. The test adopted is modified from the literature[8,9] and detects, in addition, a wide range of inorganic components such as iodides, bromides, cyanides, sulfides, thiosulfates, and nitrates. Chloride in water is determined by the red complex formed by the addition of ferric ion and mercuric thiocyanate. Use of the procedure with a spectrophotometer is described in Appendix B, p. 21.

The reagents are prepared as follows:

1. Mercuric Thiocyanate. Dissolve 0.3 gm in 100 ml methanol. Allow to stand 24 hours, filter.

2. Ferric Ion Solution. Dissolve 5 gm of ferrous ammonium sulfate, $Fe(NH_4)_2(SO_4)_2.6H_2$, in 20 ml distilled water, add 38 ml conc HNO_3; boil to oxidize to Fe^{+3} and drive off oxides of nitrogen. Make up to 100 ml.

Reagents for kit application, stored in polyethylene bottles, were unstable. Stored in glass bottles the reagents were stable; at room temperature they have been stable for over seven months. Data with this procedure are shown in Table 2 .

Kitagawa recently introduced a detector tube for chloride in water. In use the ends are snapped off the tube which is made to stand in a shallow depth of water. As water chromatographs up the tube by capillary action, a colored stain separates in the presence of chloride. The item was found insensitive and data too difficult to reproduce for quantitative work.

f. Chlorinated Compounds. Many chlorinated pesticides are detected in thin-layer chromatography by spraying them with solutions of silver nitrate or silver acetate. Following irradiation with shortwave ultraviolet light, the compounds are seen as gray to black spots[10,11]. In this study 5 ug quantities of a group of contaminants in chloroform were spotted on silica gel plates, the plates sprayed with 1% aqueous silver nitrate, and then irradiated for five minutes with shortwave ultraviolet light. Many compounds are seen as black, gray or yellow spots.

Detection of Chlorinated Compounds
With Silver Nitrate

Hazardous Materials	Response
17. Aldrin	+
18. Toxaphene	+
19. DDT	+
20. EPN	-
21. Malathion	+
22. Parathion	-
23. Dieldrin	+
24. Heptachlor	+
25. Sevin	+
26. Chlordane	+
32. 2,4-D (Acid)	-
33. 2,4,5-T (Acid)	-

A variation of this process uses bromcresol green in the silver salt reagent[12]. While variations were noted in the colors developed, there was no substantial improvement in detection capability.

Several obstacles prevent use of this method in the kit concept. Many of the relevant materials have an extremely low solubility in water; to obtain enough material for testing, extraction and concentration techniques are required. It was postulated that sufficient material could be recovered from water by shaking a 50 ml sample with 1 ml of chloroform and spotting the chloroform extracts on silica gel plates for testing. However, the poor reproducibility of extraction techniques and the complexity of operations were discouraging.

Another obstacle was the high energy requirement for the ultraviolet light. Five minute on-times periods for the ultraviolet light are excessive requirements for a power supply in the proposed field kit. The procedure was abandoned.

g. Chlorine. The Hach Free and Total Chlorine Test Kit, Model CN-66 was evaluated. The test initially appeared to have a multiple detection response. On reexamination, it was found that the readout, made with a comparator, depended on a poorly compensated reagent blank. When the tests were rerun with the colorimeter, only calcium hypochlorite gave a useful detection response.

O-Toliver (Hach No. 141), a reagent for total chlorine gave no response to the model list of contaminants and the reagent was dropped.

h. Color. Color in water was measured satisfactorily using either the colorimeter or spectrophotometer. See the data in Table 2 and procedure in Appendix B, p. 19.

i. Conductivity. A battery-operated conductivity meter, Type 70 Meter, marketed by Chemtrix, Inc., was evaluated and found generally satisfactory. The conductivity meter in the Hach DR-EL/2 kit was also tested. Results with the Hach instrument were more reproducible because it was less sensitive to the depth of immersion or motion of the probe. Data with the model list of contaminants is shown in Table 2 and the procedure is given in Appendix B, p. 18.

j. Cyanide. Detection of cyanide was studied using the Hach Cyanide Test Kit, Model CYN-3 which uses a comparator. The same reagents can be used to run the test with the colorimeter or spectrophotometer. The procedure is undesirable because it requires four individual reagents and has a 26 minute development time. Although, the test is sensitive to less than 0.01 mg/l cyanide it did not respond to a wide range of contaminants.

A recently marketed detector tube for cyanide ion in water (Kitagawa) was tested. In use the ends of the tube are snapped off and the tube made to stand in a shallow depth of water. Water rising up the tube by capillary action will elute a blue stain in the presence of cyanide. Other contaminants can elute tan to grayish colored stains. See the data in Table II and the procedure in Appendix B, p. 10 and 17. The procedure is sensitive to 5 mg/l as CN^-.

k. Detector Papers. This section is addressed to papers for detection of liquid chemical agents. (Papers impregnated with chemicals for determining pH or the presence of hardness are covered under appropriate sections). The Army uses papers impregnated with various dyes to detect liquid chemical agents in the field. Fine aerosols of the agents, on impact with the papers, dissolve a portion of the dye leaving a noticeable colored stain. The possibility of detecting organics immiscible with water using such papers was investigated. Solutions of the model list of contaminants (1000 mg/l) were shaken vigorously and spotted by pipette to M8 paper (a standard detector for liquid agent), and LAD SIN-55 and LAD SIN-26 (two advanced types of detector papers). Only nonylphenol produced noticeable red stains with the papers. No response was obtained at the 100 mg/l level. The application of the papers is too narrow for use in the kit.

l. Detector Tubes. Detector tubes are commonly used in industry to detect airborne contamination. Indeed, the Army relies heavily on these items to detect gaseous chemical agents. Detector tubes consist of a narrow glass tube containing an adsorbent, usually silica gel, held in place by porous retainers. When air is drawn through the tubes contaminants are visualized by colored reactions with chemicals impregnated on the silica gel or by drops of added reagents.

Since many of the contaminants have significant vapor pressures, an attempt at detection was made by sampling the vapors, from flasks containing 1000 mg/l concentrations of contaminants using a Bendix/Gastec benzene analyzer tube. A discoloration of the silica gel was sometimes observed,

indicative of a positive test. This finding was significant for three reasons: 1) Use of a detector designed to operate in one environment--air was extended to function in another--water. 2) This procedure detects some normally non-reactive organics in water, an area of significant weakness. 3) A large number of detector tubes for organic contaminants are readily available from many suppliers. These would constitute prepackaged detection systems equivalent to those available for inorganic contaminants.

The next effort of the investigation was to devise a means of enhancing sensitivity. One method attempted, used detector tubes to sample air which had been bubbled through the water sample to scrub out the contaminant. No significant improvement in sensitivity was found.

A convenient and effective approach made use of effervescent materials (such as Alka-Seltzer^{R} tablets) to scrub out the contaminant. In the process devised, a tablet is added to an 80 ml water sample contained in a widemouth jar. The cap, fitted with tygon tubing holding a detector kit in line is quickly screwed onto the jar. Effervescence forces the gases through the detector tube where contamination may be visualized. This procedure gave a ten-fold increase in sensitivity compared to methods in which the gases were sampled from the mouth of the flask or air was bubbled through the sample.

Findings from this study show that two basic tubes were adaptable to this process and in addition had multiple detection capabilities. One is the iodine pentoxide tube, of which, Bendix/Gastec benzene (No. 121) and toluene (No. 122) tubes are examples, and the other is the potassium dichromate tube, of which, Bendix/Gastec acetone (No. 151), ethyl acetate (No. 141) and cyclohexane (No. 103) tubes are examples. See the data (Table 2) and Appendix B, p. 10 and 16 for use of the benzene tube. The tube also responds to toluene, xylene, styrene and other organic materials.

The dichromate-type tube picks up some organic compounds not detected by the benzene tube. It would have also been included in the kit concept except that large quantities of water vapor were found to produce an interfering color response.

Studies were conducted which showed that if a scrubber tube for moisture is placed in front of the dichromate detector tube, tests are sharper, easier to read and free from the interference caused by water vapor. Scrubber tubes of the same size as the detector tube were fabricated using calcium sulfate with indicator as is commonly used in drying columns and dessicators.

Since moisture tends to fill the "openings" of silica gel, and retard detection responses, the drying tube concept is generally applicable to all detector tubes. However, since the benzene analyzer tube functions adequately without it, for simplicity, drying tubes are not included in the kit.

The following Bendix/Gastec detector tubes were tested but rejected because of lack of response or responded similarly to the iodine pentoxide or dichromate tubes: a) acrylonitrile (No. 191), b) vinyl chloride (No. 131L), c) methyl chloroform (No. 135) and d) styrene (No. 124). Kitagawa tubes: a) butadiene (No. 168b), b) ethylene (No. 108b), c) isopropyl alcohol (No. 150) and d) methyl alcohol (No. 119) were rejected for the same reasons.

In conjunction with this study, Army detector tubes for chemical agents were also tested for response to chemical pollutants. The mustard, lewisite and hydrogen cyanide tubes each detected high concentrations of phosphorous pentasulfide. In addition, the hydrogen cyanide tube also detected acetone cyanohydrin at the 10 mg/l level. The phosgene tube did not detect any of the contaminants. The military tubes were rejected because of their limited application.

m. Enzyme Systems. Detection systems for certain materials have been designed based on their ability to inhibit the action of an enzyme on a chemical substrate. The action of the enzyme on the substrate may be monitored electrometrically or in the case of colored substrates, may be measured colorimetrically.

Such systems can even be designed into simple "go-no-go" spot tests which can be assessed visually. The Army uses a detector ticket of this type with horse serum cholinesterase to detect nerve agents (Appendix B, p. 10 and 15). This device is also sensitive to certain insecticides (Table 2).

Studies were conducted using the Army enzyme test ticket in solution to obtain better color discrimination. This was achieved, but the range of response was diminished and this approach was abandoned.

The Army enzyme test uses Tris buffer, 0.05M, pH 8.0. The buffer is also used for detection of heavy metals (see Section q) and must be specially prepared. Therefore, for use in the EPA kit Army buffer was discarded and buffer described here was used:

1. Dissolve 2.43 gm of tris (hydroxymethyl) aminomethane in 100 ml distilled water.

2. Using 25 ml of the above solution, add 28.75 ml of 0.1N HCl to obtain a pH of 8.0. Dilute to a final volume of 100 ml.

3. Wash the buffer with alliquots of dithizone (diphenylthiocarbazone) solution (25 mg/l chloroform) using one part dithizone solution to 25 parts of buffer. When the dithizone layer remains blue, (about three washings) the buffer is ready for use.

A number of readily available enzyme/substrate kits for clinical application were obtained and evaluated. They were generally unresponsive and in addition, because they were intended for clinical use posed certain stability and operational limitations for field use. Included were an Ethyl Alcohol Reagent Set (Cat. No. 7919) and Alkaline Phosphatase Reagent Set (Cat. No. 7040 Code APR) from Worthington Biochemical Corporation and an Alkaline Phosphatase Reagent Set (Cat. No. 15987) and a Blood Sugar (Glucose Oxidase) (Cat. No. 15754) from Boehringer Mannheim GMBH, Biochemical Department.

n. Fluorescent Probes. A group of compounds known as fluorescent probes were investigated as general detectors for high molecular weight organic compounds[13]. Fluorescent probes are compounds which are non-fluorescent in the presence of water or other polar material, but highly fluorescent in non-polar media.

To test the probes, columns packed with macroreticular resins (Rohm and Haas, XAD-2)[14] were used to concentrate contaminants from water. Then a solution of the fluorescent probe was passed through the column so that the extracted substances would fluoresce. However, all the probes examined gave a positive fluorescent test with resin alone (a styrene-divinylbenzene copolymer) and with no contaminants present.

Contaminants were also extracted by shaking 50 ml of water sample with one ml of chloroform. The chloroform extracts were then spotted on glass fiber material, sprayed with benzene solutions of the probes and examined under longwave ultraviolet light. 7-(p-Methoxybenzylamino)-4-nitrobenz-2-oxa-1,3-diazole (MBD) at 0.0032 g/l benzene gave the best results of a number of probes tested. In many cases, a relatively high fluorescent background was noted. Further testing showed if the glass fiber was sprayed with indandione reagent (2-diphenylacetyl-1,3-indandione)[15] after application of the probe, the fluorescent background became very dull and the green fluorescent spots were more easily read. As a next step, MBD and indandione were combined into a single spray reagent at concentrations of 0.0032 g and 0.03 g respectively per liter of benzene. When the MBD-indandione method was tested against contaminants from the model list, some compounds were seen as bright green spots and others because of their ability to absorb light were seen as dull "quench" spots, both of which were considered positive tests. Materials giving either of these responses are styrene, nonylphenol, nitrophenol, EPN, malathion, parathion, sevin, fermate, 2,4-D (acid) and 2,4,5-T (acid).

It should be remembered that water prevented fluorsence of the probes. It was difficult to assure that all the water was removed after extraction with chloroform. Residual water on the glass fiber media would retard the reaction or give the effect of a false positive "quench" test. Use of small electric heaters operating off a DC power supply to remove the water became too cumbersome.

Because of false-positive tests, and need for extraction, need for a specially formulated spray reagent and an ultraviolet light, the procedure was abondoned. Elimination of the the fluorescent probe has no significant effect on detection capability since most of the contaminants (model list) are detected by other tests. It is impossible to determine if there is a serious loss in detection with respect to compounds from the Federal Register since the reaction is unpredictable.

o. Fluoride. Fluoride was determined with both the colorimeter and spectrophotometer. Detection is based on the decolorization of sodium 2-(parasulfophenylazo)-1,8-dihydroxy-3,6-naphthalene disulfonate (SPADNS), a standard test for fluoride in water[2]. A significant number of contaminants in the Federal Register listing contain fluoride. Also, the test detects other materials (see Table 2). See Appendix B, p. 22 for procedure.

p. Hardness. The Hach Total Hardness Test Kit, Model HA-71A, was used to measure hardness. The kit uses a "drop-count" titration method with ethylenediamine tetraacetate (EDTA). The end point is determined with Eriochrome Black T. It detected both calcium hypochlorite and aluminum sulfate at the 10 mg/l level, but it was not expected to have a broad detection capability and was dropped.

Test papers for hardness (Micro Essential Laboratory) were too subjective for quantitative analysis.

q. Heavy Metals. Diphenylthiocarbazone (dithizone) is a general complexing reagent commonly used for the qualitative and quanititative analysis of metals[16]. The blue reagent (chloroform solution or a similar solvent) is shaken with an aqueous solution of the metal ion; colored metal-dithizone complexes (usually pink but may be other colors) are extracted into the lower solvent layer. Adjustment of pH can make the procedure selective for specific metals. In view of the number of metals present in the Federal Register listing, a reliable heavy metals test was considered prerequisite for the kit.

The method was evaluated by shaking one ml of 0.005% dithizone in chloroform with 25 ml of water sample. Extreme variability was noted in the responses obtained. The problem was traced to the high sensitivity of the test; it was uncertain whether the reagent was reacting with pollutant or with contamination in the equipment and/or reagents. Use of individually sealed expendable plastic tubes eliminated contamination in the system. Inexpensive cellulose acetate tubes (available in bulk), were used. These soften somewhat in chloroform but not to the point of leaking. Polystyrene tubes dissolve after standing in chloroform.

Hach MercuVer™2, a dry dithizone-type reagent designed for quantitative measurment of mercury, was evaluated as a general heavy metals test. The method was simplified to a qualitative spot test. It was studied in neutral and slightly alkaline solutions. When buffered

with Tris buffer, pH 8.0, detection of multiple heavy metals was increased significantly. See Appendix B, p. 16 for the procedure developed for the kit. Also, note the section on Enzyme Systems (Section m) for the preparation and washing of Tris buffer with dithizone for this application.

Response to contaminants from the model list (Table 2) does not show the capability of this reagent to heavy metals. Additional solutions of metal compounds were prepared for further evaluation. Positive tests were obtained with 100 mg/l or less of the following heavy metals: cadmium, cobalt, copper, gold, lead, mercury, silver, and zinc.

A recent report[17] described the preparation of open-cell polyurethane cubes impregnated with chromogenic organic reagents. It was conceived that the expendable cellulose acetate tubes could be prepackaged with dithizone impregnated foam cubes. In use the cube filled tubes would require only the addition of water, shaking, and observation for a change in the color of the cube.

Polyurethane was washed and prepared according to the reported procedure. Cubes about 1 cm on the side were placed in polyethylene bags and kneaded until uniform in color with solutions of dithizone in various phthalates (3 ml solutions to 1 gm of cubes was used). The most stable preparation was found to be 0.025 gm of dithizone in 50 ml of diisobutyl phthalate.

Tests run with dithizone foam cubes showed them to be as sensitive as MercuVer™2 even without the use of Tris buffer. Unfortunately, most of the preparations showed serious signs of deterioration after several months at room temperature. Although there is some possibility of stabilizing the reagent cubes with ascorbic acid or other antioxidants, time limitations prevented completion of this study.

r. Ion-Selective Electrodes. Several ion-selective electrodes were obtained to assess their feasibility for the detector kit. An ammonia electrode (Chemtrix) was evaluated in conjunction with the Chemtrix Type 40E battery-operated portable pH meter which was then the current choice of meters. Although supposedly compatible, the ammonia electrode never functioned satisfactorily, apparently due to limitations of the meter.

A divalent ion-selective electrode (Corning No. 476235) with a laboratory type meter was tested against the contaminants (model list). The electrode was prepared as described in the manufacturer's instructions and immersed in each stirred solution along with a calomel reference electrode. After equilibration the potentials were read and recorded. The concentrations of the contaminants was 1000 mg/l or a saturated solution whichever was less. The divalent electrode behaved much like a conductivity electrode responsive to total ionic strength of the solution. Results were found to follow a definite trend. Distilled water read a potential of -100 mV. The non-ionic organics all showed a reading of -110 to -129 mV. Sevin at -135 and

nitrophenol at -140 mV were the only exceptions. The ionic inorganics all gave readings of -60 to -90 mV. The only significant departures were phosphorus pentasulfide and aluminum sulfate at 0 mV and chlorosufonic acid at +35 mV.

A redox electrode which was considered was found unsuitable; the presence of both the oxidized and reduced form of the substance to be detected was required to obtain a signal. This necessitates polarizing the electrode which in turn requires an additional regulated power supply for use and calibration. Also, the platinum surface is not reproducible from day-to-day and would need continued standardization.

All-in-all work with electrodes was not promising. They pose a number of calibration problems and do not appear to be sufficiently versatile for non-routine situations encountered in the field. In addition, they show limited capability for organics which is the area of prime concern. Also, electrodes are expensive and require the use of a significantly more expensive meter.

s. Methylene Blue Active Substances (MBAS). This method is used to determine alkyl benzene sulfonate (ABS) in water. Under the conditions specified in the test procedure, substances which react are referred to as methylene blue active substances[2]. It measures surfactants in water. The procedure is subject to interferences such as organically bound sulfates, sulfonates, carboxylates, phosphates, phenols, cyanides, thiocyanates and even some inorganic ions like nitrate and chloride[4]. It was the long list of interferences which led to its consideration as a multiple response detector.

The original method is designed for quantitative measurements using a colorimeter. For simplicity and to reduce the volume of reagents, the procedure was evaluated for use in a spot test application. In use, a buffered aqueous solution of methylene blue is mixed with the water sample in a test tube and shaken with 1 ml of chloroform. Materials in the water phase which are active with methylene blue extract into the solvent phase as blue colored products.

The procedure did respond to phosphorus pentasulfide, acetone cyanohydrin, xylenes, nitophenol, ammonium nitrate, malathion, parathion, fermate, 2,4-D (acid) and 2,4,5-T (acid). However, because response was obtained only at the highest concentration and the intensity of the color was not very strong the test was considered too unreliable for the kit.

t. Nitrate Nitrogen. Hach Nitrate Test Kit, Model NI-11 was evaluated for nitrate in water. The kit which uses prepackaged reagents and a color comparator was rejected in favor of the method used with the colorimeter or spectrophotometer. See Table 2 for results with the contaminants (model list) and Appendix B, p. 19 for the procedure. It is seen that the method is responsive to materials other than nitrate ion.

u. Odor. Tests were conducted by simply sniffing the mouths of flasks containing the different dilutions of contaminants in distilled water. Odor was easily one of the most sensitive detection means with broadest response capability. The following materials were detectable at the concentrations noted by two or more investigators:

Detection Sensitivities of Contaminants by Odor, mg/l

Contaminant	Detection Limit
Phenol	1000
Benzene	1000
Phosphorus Pentasulfide	1
Styrene	0.7
Acetone Cyanohydrin	100
Isoprene	0.1
Xylenes	1.3
Nitrophenol	100
Aluminum Sulfate	1000
Toxaphene	3
EPN	5
Malathion	1.5
Parathion	0.3
Heptachlor	0.02
Chlordane	0.1
Fermate	120

Use of odor as an analytical tool is difficult because it is extremely subjective and impossible to standardize in a practical manner for use in the field by different operators. Use of odor as a detector is further complicated by the fact that airborne vapors of contaminant would make it impossible to pinpoint its location in the water. Use of odor as a detection method was rejected.

v. pH. It was decided that pH monitoring would be important in tracing contamination and therefore accuracy and sensitivity were considered prerequisite. Impregnated papers and chemical indicators with comparators were too subjective. Several electronic meters were considered. A compact battery-operated pH meter, Type 40E marketed by Chemtrix, provided reliable and accurate measurements. A similar meter by Hach, Model 1975 pH meter, appeared to give quicker responses with more reproducible results. The Hach meter, however, was selected only for convenience in design (see Section 5).

Combination pH electrodes were studied with both meters and were satisfactory. A sealed combination pH electrode marketed by Chemtrix requiring no servicing was also tested. The unit was convenient in that it was ruggedized, expendable and had a life of two years. However, response with it was sluggish and generally slower than with the conventional combination electrodes.

See Table 2 for the pH data; Appendix B, p. 9 and 18 describes pH measurement in the field.

w. Phenolics. Commercial kits for phenol use the 4-aminoantipyrene method which involves a lengthy and awkward extraction procedure. An extremely sensitive test for phenol based on the Berthelot reaction[18,19,20] was studied for use with the Hach DR Colorimeter or Spectrophotometer. The reagents required and the procedure developed is described:

1. Reagents: Bleach/Caustic - Dilute 10 ml commercial bleach (5% NaOCl) + 60 ml 10% aqueous NaOH to 1 liter with distilled water.

Ammonia Reagent - Dilute 10 ml conc. ammonium hydroxide to 100 ml with distilled water.

Sodium Nitroprusside - Dissolve 6.0 g of sodium nitroprusside in 100 ml distilled water. Dilute 12.5 ml of this solution to 500 ml with distilled water to make a working solution.

2. Procedure: Place 21 ml water sample in a glass-stoppered 25 ml graduate. Add 2 ml Bleach/Caustic, 1 ml Ammonia Reagent and 1 ml working solution of Sodium Nitroprusside. Run a blank in parallel. Incubate the sample and blank for 5 minutes in a water bath at 56 $\pm$ 2°C. Cool at room temperature for 5 minutes. Read in the DR Colorimeter using the 2408 filter or in the spectrophotometer at a wavelength of 625 nm. Compare with phenol standards prepared in a similar manner (Heating for field application was accomplished using a canned dry heat).

Although extraction procedures are avoided, the method is lengthy and it introduced a need for heat and a temperature controlled water bath. The method was found vey useful as a laboratory procedure but unsuitable for field work.

A phenol spot test was developed by modifying the quantitative Hach procedure. The developed method sacrifices quantification for simplicity and reduction of reagent volume. See Table 2 for the data and Appendix B, p. 17 for the procedure.

Several experiments were conducted to prepare phenol test devices similar to the cellulose acetate devices containing dithizone cubes for heavy metals. A solution was prepared containing 1 g 4-aminoantipyrene and 1 g tris (hydroxymethyl) aminomethane in 5 ml dimethylphthalate-acetone (1:1). This solution was kneaded with 1 g urethane cubes[17] in a plastic bag to obtain a uniform yellow color. The cubes sprinkled with 5-10 crystals of potassium ferricyanide are ready for use. The items were found to give quick and easily discernible tests with solutions containing as little as 0.1 mg/l phenol. Sufficient time was not available at the end of the program to determine the stability of the system.

x. Phosphate. Hach Total Phosphate Test Kit, Model PO-24, was evaluated for detection of phosphate in water. The kit has prepackaged reagents and a color comparator. Results were more reliable when the same reagents are used with the Hach DR Colorimeter or Hach Spectrophotometer. See data, Table 2 and procedure, Appendix B, p.20. Additional experiments in support of the "paper analysis" showed that the procedure would also detect concentrations of less than 100 mg/l of arsenates, arsenites, and bromates.

y. Sulfate. Hach Sulfate Test Kit, Model SF-1, was evaluated for detection of sulfate. The kit uses prepackaged reagent which causes a turbidity in the presence of sulfate ion. Concentration is correlated to the ability to observe a heavy "X" marking at the bottom of a graduate through different depths of the water sample with the turbidity. The procedure is extremely insensitive and can only be considered semiquantitative.

The same reagent with either the colorimeter or spectrophotometer gives a 10-fold increase in sensitivity with greater reliability. The procedure is highly specific; it was adopted for the kit in spite of this fact due to the large number of sulfate compounds in the Federal Register listing. See Table II for the data; the procedure is given in Appendix B, p. 20.

z. Turbidity. Turbidity was measured using both the colorimeter and spectrophotometer. Tests were conducted only to show that turbidity can be measured and could be indicative of insoluble contamination. Because the physical form of contaminants (e.g. fine powders, flakes, granules, emulsions, etc.) cannot be anticipated, nor can the conditions of water environment (e.g. turbulent creek, quiet pond, etc.) no attempt was made to correlate turbidity caused by the contaminants (model list) to contamination.

See Appendix B, p. 18 for the procedure adopted for the kit.

TESTING WITH NATURAL WATERS

The efficacy of the selected procedures was determined with water samples taken from Winter's Run, Big Gunpowder Falls and the Susquehanna River in Harford County, MD. The waters were analyzed as received using the candidate methods. Afterwards the samples were polluted in the laboratory with contaminants from the model list in the manner described previously. The detection limit was determined for the qualitative spot tests; the lowest quantity of contaminant required to produce a significant change over that of the original water was determined for the quantitative tests. Usually, there was some loss in sensitivity when compared to distilled water; however, nothing significant was found to preclude use of these methods in a detector kit.

The data is summarized in Tables 3, 4 and 5.

Table 2
Detection Systems for Hazardous Materials
(Concentrations of Hazardous Materials in mg/l showing a measureable response)

Hazardous Materials	Chol. Inhib.	Benzene	Heavy Metals	Phenol	Cyanide	pH	Conduct.	Nitrate N_2	Color	Sulfate	Phosphate	Ammonia N_2	Chloride	Fluoride
1. Phenol				1										
2. Methyl Alcohol														
3. Acrylonitrile														
4. Chlorosulfonic Acid	1000				1000	1	10			10			10	1000
5. Benzene		1												
6. Ammonium Chloride							100					1	10	
7. Phosphorus Pentasulfide		100			100	1	100	10			1	1	10	10
8. Styrene		6.6						660					660	
9. Acetone Cyanohydrin					100			100					1	
10. Calcium Hypochlorite			1000			1	100					1000	10	
11. Nonylphenol				1.7		17								
12. Isoprene		1						10					100	
13. Xylenes		1.3												
14. Nitrophenol					10	100			1			1	1000	
15. Ammonium Nitrate						1000	100	10				1		
16. Aluminum Sulfate						10	1000			10				1000
17. Aldrin														
18. Toxaphene						3								
19. DDT														
20. EPN	500					50			0.5			50	500	
21. Malathion	14.5	145				145						145	14.5	
22. Parathion	2.5											2.5	25	
23. Dieldrin	0.3											0.03		
24. Heptachlor	0.2											0.02	0.2	
25. Sevin	4			0.04				0.4				0.4	4	
26. Chlordane														
27. Fermate		12			120	12		12)	120			12	12	120
28. Lead Arsenate			2.5			0.3						2.5		
29. Disodium Methylarsenate						10	1000				2.5			1000
30. Phenylmercuric Chloride			6.9									0.7		
31. 2,4-D (Acid)		90				90								
32. 2,4,5-T (Acid)		280				28	280							
33. Ammonium Phosphate, Dibasic			1000			10	100				1	1		100

NOTE: Instrumental measureable responses are indicated by a change of 0.5 pH unit, gain in conductivity of 50 μmhos/cm or change of transmittance of 5%.

Table 3

Detection of Contaminants in Water from Winter's Run

(Concentrations of Hazardous Materials in mg/l showing a measureable response)

Hazardous Materials	Chol. Inhib.	Benzene	Heavy Metals	Phenol	Cyanide	pH	Conduct.	Nitrate N_2	Color	Sulfate	Phosphate	Ammonia N_2	Chloride	Fluoride
1. Phenol				1										
2. Methyl Alcohol														
3. Acrylonitrile														
4. Chlorosulfonic Acid	1000				1000	10	10			10			10	1000
5. Benzene		1												
6. Ammonium Chloride							100					1	10	
7. Phosphorus Pentasulfide		100			100	10	100	10			1	1	10	100
8. Styrene		6.6												
9. Acetone Cyanohydrin					100			1000					10	
10. Calcium Hypochlorite			1000			1	100					1000	100	
11. Nonylphenol				1.7										
12. Isoprene		10						10						
13. Xylenes		1.3												
14. Nitrophenol					10	100			10			1	1000	
15. Ammonium Nitrate							100	10				1		1000
16. Aluminum Sulfate						100	1000			10				
17. Aldrin														
18. Toxaphene														
19. DDT														
20. EPN	500								5			500		
21. Malathion	14.5	145				145						145	145	
22. Parathion	2.5								2.5			2.5		
23. Dieldrin												0.03		
24. Heptachlor												0.02		
25. Sevin	4			0.4								4	40	
26. Chlordane														
27. Fermate		12			120			120				120	120	
28. Lead Arsenate											2.5	2.5		
29. Disodium Methylarsenate						100	1000							1000
30. Phenylmercuric Chloride			6.9									6.9		
31. 2,4-D (Acid)		90				900								
32. 2,4,5-T (Acid)		280				280								
33. Ammonium Phosphate, Dibasic			1000			10	100				1	1		10

NOTE: Instrumental measureable responses are indicated by a change of 0.5 pH unit, gain in conductivity of 50 μmhos/cm or change of transmittance of 5%.

Table 4
Detection of Contaminants in Water from Big Gunpowder Falls
(Concentrations of Hazardous Materials in mg/l showing a measureable response)

Hazardous Materials	Chol. Inhib.	Benzene	Heavy Metals	Phenol	Cyanide	pH	Conduct.	Nitrate N_2	Color	Sulfate	Phosphate	Ammonia N_2	Chloride	Fluoride
1. Phenol				1										
2. Methyl Alcohol														
3. Acrylonitrile														
4. Chlorosulfonic Acid	1000				1000	10	10			10			10	1000
5. Benzene		1												
6. Ammonium Chloride							100					1	10	
7. Phosphorus Pentasulfide		100			100	10	100	100			10	1	10	100
8. Styrene		6.6												
9. Acetone Cyanohydrin					100			100					10	
10. Calcium Hypochlorite			1000			100	100					1000	100	
11. Nonylphenol				1.7					10					
12. Isoprene		10												
13. Xylenes		1.3												
14. Nitrophenol					10	100						1	1000	
15. Ammonium Nitrate							100	10				1		
16. Aluminum Sulfate						100	1000			10				1000
17. Aldrin														
18. Toxaphene														
19. DDT														
20. EPN									500			500		
21. Malathion		145				145						145	145	
22. Parathion	2.5								2.5			25		
23. Dieldrin												0.03		
24. Heptachlor												0.02		
25. Sevin	4			4								40	40	
26. Chlordane														
27. Fermate		12			120			12				120	120	
28. Lead Arsenate						2.5					0.3	2.5		
29. Disodium Methylarsenate						100	1000							1000
30. Phenylmercuric Chloride			0.07									0.7		
31. 2,4-D (Acid)		900				900								
32. 2,4,5-T (Acid)		280				280	280							
33. Ammonium Phosphate, Dibasic						10	100				1	1		10

NOTE: Instrumental measureable responses are indicated by a change of 0.5 pH unit, gain in conductivity of 50 μmhos/cm or change in transmittance of 5%

Table 5

Detection of Contaminants in Water from Susquehanna River

(Concentrations of Hazardous Materials in mg/l showing a measureable response)

Hazardous Materials	Chol. Inhib.	Benzene	Heavy Metals	Phenol	Cyanide	pH	Conduct.	Nitrate N_2	Color	Sulfate	Phosphate	Ammonia N_2	Chloride	Fluoride
1. Phenol				1										
2. Methyl Alcohol							10							
3. Acrylonitrile	1000				1000	10				10			10	1000
4. Chlorosulfonic Acid		1					100					1	10	
5. Benzene							100							
6. Ammonium Chloride		100			100	100		100			1	1	10	10
7. Phosphorus Pentasulfide		6.6												
8. Styrene					100			100					10	
9. Acetone Cyanohydrin			1000			100	100					1000	100	
10. Calcium Hypochlorite				1.7										
11. Nonylphenol		1												
12. Isoprene		1.3												
13. Xylenes					10	100			10			1	1000	
14. Nitrophenol							100	10				1		
15. Ammonium Nitrate						100	1000			10				1000
16. Aluminum Sulfate														
17. Aldrin														
18. Toxaphene														
19. DDT									500			500		
20. EPN						145						145	145	
21. Malathion	145	145							25			2.5		
22. Parathion	2.5											0.3		
23. Dieldrin														
24. Heptachlor												40	40	
25. Sevin	4			0.4										
26. Chlordane														
27. Fermate		12			120			12				12	12	
28. Lead Arsenate			2.5			2.5					0.3			
29. Disodium Methylarsenate						100	1000							1000
30. Phenylmercuric Chloride			0.07									0.7		
31. 2,4-D (Acid)		90				90								
32. 2,4,5-T (Acid)		280				280	280							
33. Ammonium Phosphate, Dibasic						1000	100				1	1		100

NOTE: Instrumental measureable responses are indicated by a change of 0.5 pH unit, gain in conductivity of 50 μmhos/cm or change in transmittance of 5%

SURVEY OF MARYLAND WATERS

Natural waters from north central Maryland were sampled to determine range of properties and concentrations of natural constituents.

Twenty-two water sampes were taken from a loop which reached from approximately 10 miles north of Baltimore, northwest to Emmitsburg, south toward Frederick, south-easterly towards Laurel, around Baltimore and back to five miles south of the starting point. The names of the waters where known were noted; others were identified by their location. The selected detection methods were used to characterize the waters and to anticipate any problems which might arise in use of the kit. No difficulties were experienced in analysis. Generally speaking the waters were of high quality and changes in them due to contamination should be easy to detect. Only when samples were taken near the city of Baltimore, in the harbor area, did some of the background readings (e.g. conductivity, sulfate and chloride) get so high as to present a significant background.

Data are summarized in Table 6.

Table 6

Background of Natural Waters of North Central Maryland

	Water Source	Turbidity, FTU	Chol. Inhib.	Benzene	Heavy Metals	Phenol	Cyanide	pH	Conduct. umhos/cm	Nitrate, N_2 mg/l	Color APHA Units	Sulfate mg/l	Phosphate mg/l	Ammonia, N_2 mg/l	Chloride mg/l	Fluoride mg/l
1.	Loch Raven Reservoir	10	-	*	-	-	-	6.9	139	4.8	20	3	<0.1	<0.1	10.4	0.1
2.	Liberty Reservoir	10	-	*	-	-	-	6.9	136	6.2	40	4	<0.1	0.7	15.9	0.1
3.	Stream West of Taneytown	10	-	*	+	-	-	7.1	165	7.5	30	7	<0.1	<0.1	9.1	0.1
4.	Stream West of Taneytown	10	-	*	-	-	-	7.1	135	5.7	30	8	<0.1	0.4	8.2	0.2
5.	Stream West of Taneytown	10	-	*	+	-	-	7.2	249	4.8	40	18	0.3	0.8	25.4	0.2
6.	Monacacy River, Rt 97	10	-	*	+	-	-	7.1	185	3.8	50	16	0.2	0.6	10.4	0.3
7.	Stream East of Emmitsburg	10	-	*	-	-	-	7.4	165	2.2	40	12	<0.1	<0.1	5.0	0.2
8.	Stream South of Emmitsburg	10	-	-	-	-	-	7.1	104	1.8	10	4	<0.1	<0.1	4.0	0.2
9.	Stream North of Frederick	10	-	-	-	-	-	6.8	60	0.0	10	1	<0.1	<0.1	1.3	0.1
10.	Stream North of Frederick	10	-	-	-	-	-	7.1	160	7.0	10	11	<0.1	<0.1	5.5	0.2
11.	Monacacy River, Rt 80	10	-	-	-	-	-	7.0	200	7.5	30	15	0.2	0.3	8.8	0.2
12.	Hawlings Run	26	-	-	-	-	-	6.8	78	5.3	20	1	<0.1	<0.1	6.0	0.1
13.	Pautuxent River, Rt 97	10	-	-	-	-	-	6.9	106	4.8	20	1	<0.1	<0.1	6.4	0.1
14.	Cattail Creek	10	-	-	-	-	-	6.8	110	6.2	10	1	<0.1	<0.1	9.1	0.1
15.	Pautuxent River, Rt 108	10	-	-	-	-	-	6.9	95	4.0	0	3	<0.1	<0.1	7.8	0.2
16.	Stream Near Brinklow	10	-	-	-	-	-	6.8	84	3.7	10	3	<0.1	<0.1	7.8	0.1
17.	Middle Pautuxent River	10	-	*	-	-	-	7.3	130	4.8	0	6	<0.1	<0.1	13.4	0.1
18.	Little Pautuxent River	10	-	-	-	-	-	7.2	142	4.0	0	8	<0.1	<0.1	17.7	0.2
19.	Patapsco River, Rt 144	10	-	*	-	-	-	7.3	113	4.8	0	7	<0.1	0.3	12.5	0.2
20.	Patapsco River, Baltimore	10	-	*	-	-	-	6.6	162	3.8	0	12	<0.1	1.6	16.5	0.1
21.	Patapsco River, Baltimore	10	-	-	-	-	-	7.3	600	3.7	30	35	0.4	0.5	110.0	0.2
22.	Falls Run	10	-	-	-	-	-	7.5	165	4.0	0	5	<0.1	<0.1	15.0	0.2

*Stains were less than 1-2mm, tests considered negative.

NOTE: All samples represent different sources of water, or significantly different points of the same body of water.

SECTION 5
FORMULATION OF DETECTION CONCEPT

SELECTED TESTS

Many of the reasons for selection or rejection of individual tests have been discussed (Experimental Studies). Unfortunately, selection is often based not necessarily on capability required, but on what is available. All-in-all every effort was made to assemble the best combination of tests from what is available, with emphasis placed on reaching previously set goals of the detector kit. Generally, procedures which are simple, reliable, stable and commercially available (when possible) and lend themselves to the detection concept were selected. To maintain compactness, duplication of detection capability was avoided. For example, numerous metal tests were eliminated by adopting dithizone as a general heavy metals test.

A summary of characteristics of the 15 procedures selected for the kit are given in Table 7 .

PROJECTED DETECTION CAPABILITY

A "paper analysis" was made to project the probability of detection of the 370 compounds listed in the Federal Register. Each compound was screened by comparison with the detection tests (except turbidity) listed in Table 7 ; that is, each compound was compared with a) data derived from laboratory testing with contaminants from the model list, and b) data derived from handbooks. A compound was considered detectable by a particular test if it was judged that a saturated solution or 1000 mg/l of the compound (whichever was less) has a significantly greater than 50% chance of responding. Where information was considered inadequate to make a satisfactory judgment, individual additional testing was considered. (For example, in this manner it was determined that

TABLE 7
Summary of Methods Selected for Detector Kit

Detection Parameter	Type*	Process	Reagents**	Time, Min.	Types of Substances Detected	Max. Sens. in Nat. Water, mg/l
Cholinesterase Inhibitors	Qualitative	Enzyme Ticket	M30A1, Chemical Agent Detector Kit	< 8	Certain insecticides, very acid materials.	< 3
Benzene	Qualitative	Detector Tube	Bendix/Gastec	< 2	Benzene, styrene, xylene, toluene, other organic materials.	1
Heavy Metals	Qualitative	Extraction/ Test Tube	Hach/Laboratory	< 1	Many metals. Also some organic materials.	< 10
Phenol	Qualitative	Extraction/ Cent. Tube	Hach	< 1	Phenolics.	1
Cyanide	Qualitative	Detector Tube	Kitagawa	< 1	Cyanide ion. Certain other organic and inorganic materials.	10
pH	Quantitative	Meter	—	< 1	Acidic or basic organic and inorganic materials.	< 3
Conductivity	Quantitative	Meter	—	< 1	Inorganic compounds and other materials with high ionic strength.	10
Nitrate Nitrogen	Quantitative	Spectro-photometer	Hach	< 6	Nitrate ion. Certain other organic and inorganic substances.	10
Color	Quantitative	Spectro-photometer	--	< 1	Highly colored organic or inorganic substances.	< 5
Sulfate	Quantitative	Spectro-photometer	Hach	< 10	Sulfate ion.	10
Phosphate	Quantitative	Spectro-photometer	Hach	< 2	Phosphate ion. Certain arsenates arsenites and bromates.	< 1
Ammonia Nitrogen	Quantitative	Spectro-photometer	Hach	< 10	Wide range of organic and inorganic ammonia containing materials.	< 1
Chloride	Quantitative	Spectro-photometer	Laboratory	< 10	Wide range of sulfides, bromides, cyanides, sulfides, thiosulfates, chlorides.	10
Fluoride	Quantitative	Spectro-photometer	Hach	< 2	Fluoride. Various inorganic and organic materials.	10
Turbidity	Quantitative	Spectro-photometer	--	<1	Low solubility materials.	Not determined

* Quantitative in this context means that a numerical value is assigned to the data.

** Laboratory indicates reagents which are specially prepared.

nitrites would respond to the nitrate test and sulfites were detected by the sulfate test, whereas, thiosulfates were not. Also, certain arsenates, arsenites and bromates respond to the phosphate test, but chromate does not). Extensive testing was conducted with heavy metals and ammonia containing compounds. These data have already been reported under appropriate sections in Experimental Studies.

Highlights of the "paper analysis" are given in Table 8 Data from this study indicate that 85% of the contaminants would respond to at least one parameter; 58%, 30% and 10% would respond to two, three, and four or more parameters respectively. (See complete data in Appendix A.)

Because certain compounds don't show responses for the detection parameters doesn't necessarily mean non-detection. Rather, many of these structures are very complex and their reactivity cannot be predicted. Judging by some of the unexpected responses obtained with many of the detection methods it is to be expected that some of these materials are detectable by present methods.

Table 8

Probability of Detection of Water Contaminants

(Based on nominally 370 compounds)

Detection Parameter	Probable Responses
Cholinesterase Inhibitors	16
Benzene	20
Heavy Metals	102
Phenol	12
Cyanide	11
pH	66
Conductivity	201
Nitrate Nitrogen	18
Color	50
Sulfate	23
Phosphate	18
Ammonia Nitrogen	58
Chloride	72
Fluoride	24

USE CONCEPT

Our goal was to develop a field kit using a standardized system so that results are easily obtained and interpreted by a senior technician. This goal, generally, has been met; tests are easy to perform, short and designed for ease of operation in the field. However, detecting almost any contaminant in any water background is a formidable task which requires ingenuity and the interpretive skills of a dedicated investigator.

The detection concept relies basically on three evaluations as shown:

DETECTION CONCEPT

General Assessment	Relative Measurements
1. Appearance	9. pH
2. Color of Filtered Water	10. Conductivity
3. Color of Suspended Matter	11. Turbidity
	12. Nitrate Nitrogen
Spot Tests	13. Color
4. Cholinesterase Inhibitors	14. Sulfate
5. Benzene	15. Phosphate
6. Heavy Metals	16. Ammonia Nitrogen
7. Phenol	17. Chloride
8. Cyanide	18. Fluoride

General Assessment procedures allow the operator to make a general evaluation of the water to be tested. That is, are there slicks present? -- is the water colored? -- is there a lot of suspended material?-- is the suspended material colored? Spot Tests are used to indicate if a particular type of contaminant is present. A positive response to any of these tests should be considered contamination since it would indicate compounds not normally found or desired in water. The final group of tests, Relative Measurements, is based on constituents of natural water. Contamination is indicated by a significant increase in one of these parameters compared to uncontaminated water.

To use the detection concept the investigator must assess two major factors: a) The nature of the water source -- is it stationary, like a lake or pond, or is it moving like a stream or river? b) The nature of the contaminant -- is it specifically known or partially known, such that it may be acid or base, organic or inorganic, phosphate containing, colored, etc?

In situations where the contaminant is unknown, the investigator confronts the situation in an orderly fashion: (1) He immediately conducts the General Assessment evaluations. (2) He considers using the Spot Tests since these are simple to use and easy to evaluate. (3) He proceeds to the Relative Measurements. If the location of the contaminant is based on a relative measurement then he must make a comparison between the area of

suspected contamination and a clean source of water (reference sample). In the case of a stream, the reference sample may be taken at a location upstream from the contamination. If the body of water is a lake, he must use his best judgment and select an area believed to be clean. Once the reference sample is established, it is a matter of deciding which relative tests can be used in detecting significant changes.

In cases where the specific contamination is known, the investigator proceeds directly to the applicable tests. For example, an investigator confronted with a spill of phosphoric acid would immediately perform pH, conductivity and phosphate tests and determine which is most suitable for this situation.

A special data sheet for recording and reporting of contamination has been designed to correspond with the detection concept (see Appendix B, p. 10).

DESIGN AND FABRICATION OF PROTOTYPE KITS

A significant part of design was concerned with human factors considerations, i.e. man/machine interfaces. The purpose is to select procedures and instrumentation which make tests easy to perform and evaluate and which provide reliable information. Some of these factors have already been considered when procedures were selected. For example, use of test papers, color comparators and pH indicators are considered too subjective, particularly when measurements must be made relative to a reference sample. Use of a quality pH meter, conductivity meter and colorimeter is a prerquisite. Spot tests, which are also subjective, are adopted for use in the detection concept as gross tests to be used only on a "go no-go" basis.

Some of the instruments considered for this program (e.g. the Chemtrix Type 40E pH meter and Chemtrix Type 70 conductivity meter) have already been discussed. Much of the development data was obtained with the instruments; both meet the level of quality and performance required for the kit. Also, the Hach DR Colorimeter was used extensively and was found to be a highly satisfactory portable colorimeter.

The Mini-Spec 20 Spectrophotometer (marketed by Bausch and Lomb, Analytical Systems Division) was also considered briefly for the kit. A demonstration of the Mini-Spec 20, which is a full grating spectrophotometer about the size of a pocket calculator, showed it to be a sensitive, accurate and versatile instrument. The feeling, however, is that it may be too delicate and sophisticated for field use; many of the pieces used are small and do not appear to easily lend themselves to operation under rugged conditions.

Originally it was planned to integrate the best available instrumentation into a suitable carrying case. A prime concern all-along was to restrict kit size and weight to maintain the man-portable concept. When it appeared the final package might become too large and unwieldy, instruments combining

several functions were sought. In fact, at that time, a 4 Function Water Analyzer (International Ecological Systems) was on the market. The instrument, which measured about 5x10x11 inches provided pH, conductivity, colorimeteric and thermal measurements. This item was considered until its continued availability, service and replacement parts became uncertain.

Finally, a decision was made to adapt the DR-EL/2 portable test kit marketed by Hach which incorporates a spectrophotometer and a conductivity meter. Hach agreed to provide this item without the standard equipment or reagents. In addition, a Hach Model 1975 battery-operated pH meter was dismantled and reassembled into a special compartment fabricated into the lid of the case; when the case is open the pH meter faces the operator. The battery for the pH meter is next to the power supply for the spectrophotometer and conductivity meter.

A significant human factors advantage is gained by use of the spectrophotometer instead of a filter colorimeter since color filter changes are not required for the different colorimetric analyses.

It was originally planned to use a case meeting military specifications for the detector kit. However, the Hach case by comparison, while not as rugged, is considerably lighter and less expensive. Also use of the Hach case significantly reduces special design and fabrication costs for the kit.

A particularly important piece of equipment included in the kit is a plastic syringe with stainless steel head fitted with a glass fiber disc to filter the water sample. It is used for general assessment of the water sample and to process it for colorimetric analysis.

Other necessary ancillary equipment or reagents were purchased from available sources or prepared in the laboratory. Many of the purchased reagents and equipment were repackaged in the kit in the interest of economy of space. It must be remembered that only the detector tickets and substrate are used from the M30A1 Chemical Agent Detector Kit. The Tris buffer is prepared in the laboratory (Experimental Studies, m. Enzyme Systems) since it must be washed with dithizone for use in the heavy metals test.

An Operator's Manual (Appendix B) was prepared for use with the kit. It describes in detail the use concept, equipment and procedures used. A source listing for consumable parts is given. Hach manuals for the DR-EL/2 portable test kit and Model 1975 pH meter are also provided. In addition, laminated outlines of procedures are included for use in the field.

Tests were conducted with the kits to demonstrate their fieldability. Modifications were made to the Operator's Manual as a result of these exercises. The detector kits resulting from this program are shown in Figures 1 and 2.

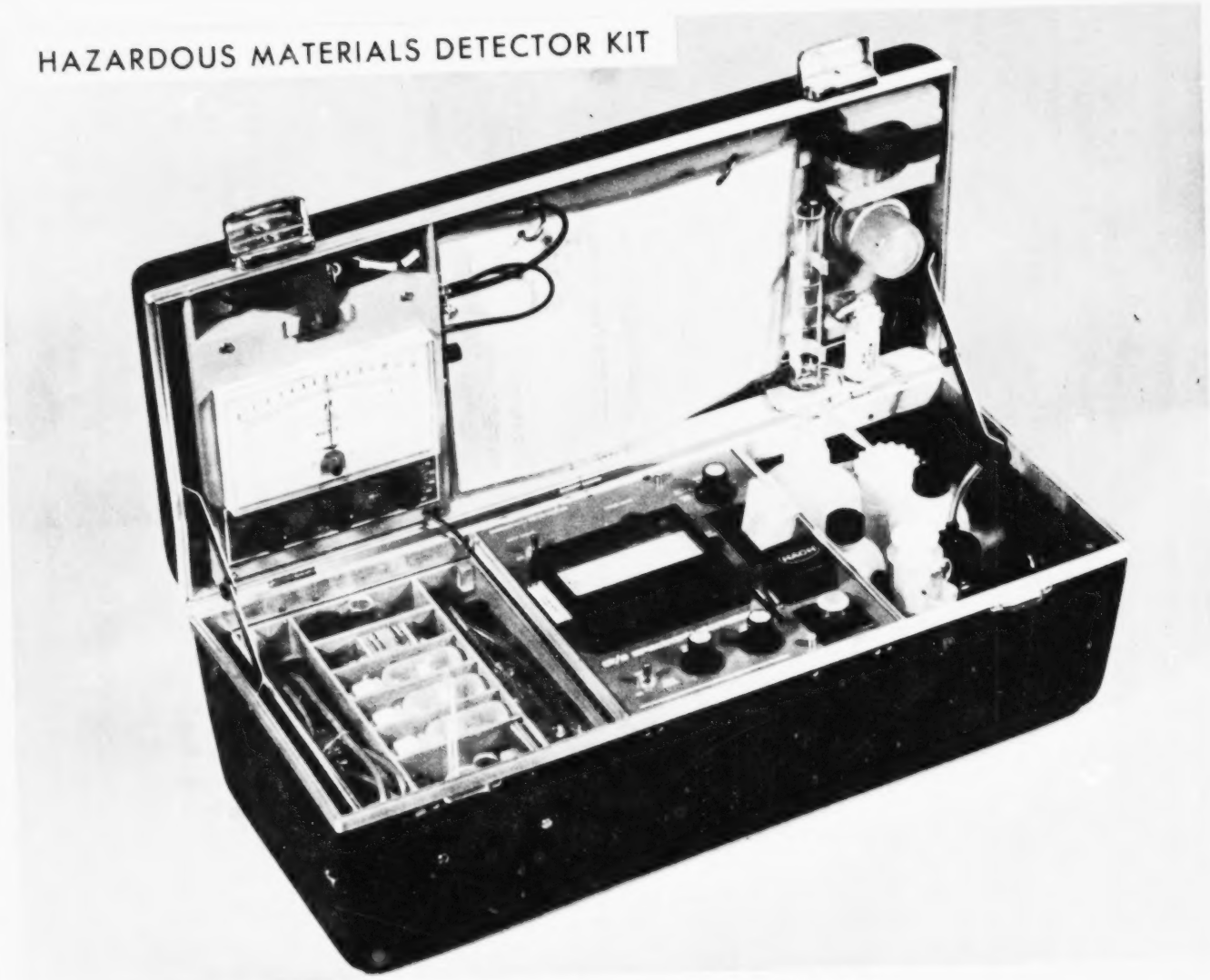

Figure 1 Hazardous Materials Detector Kit

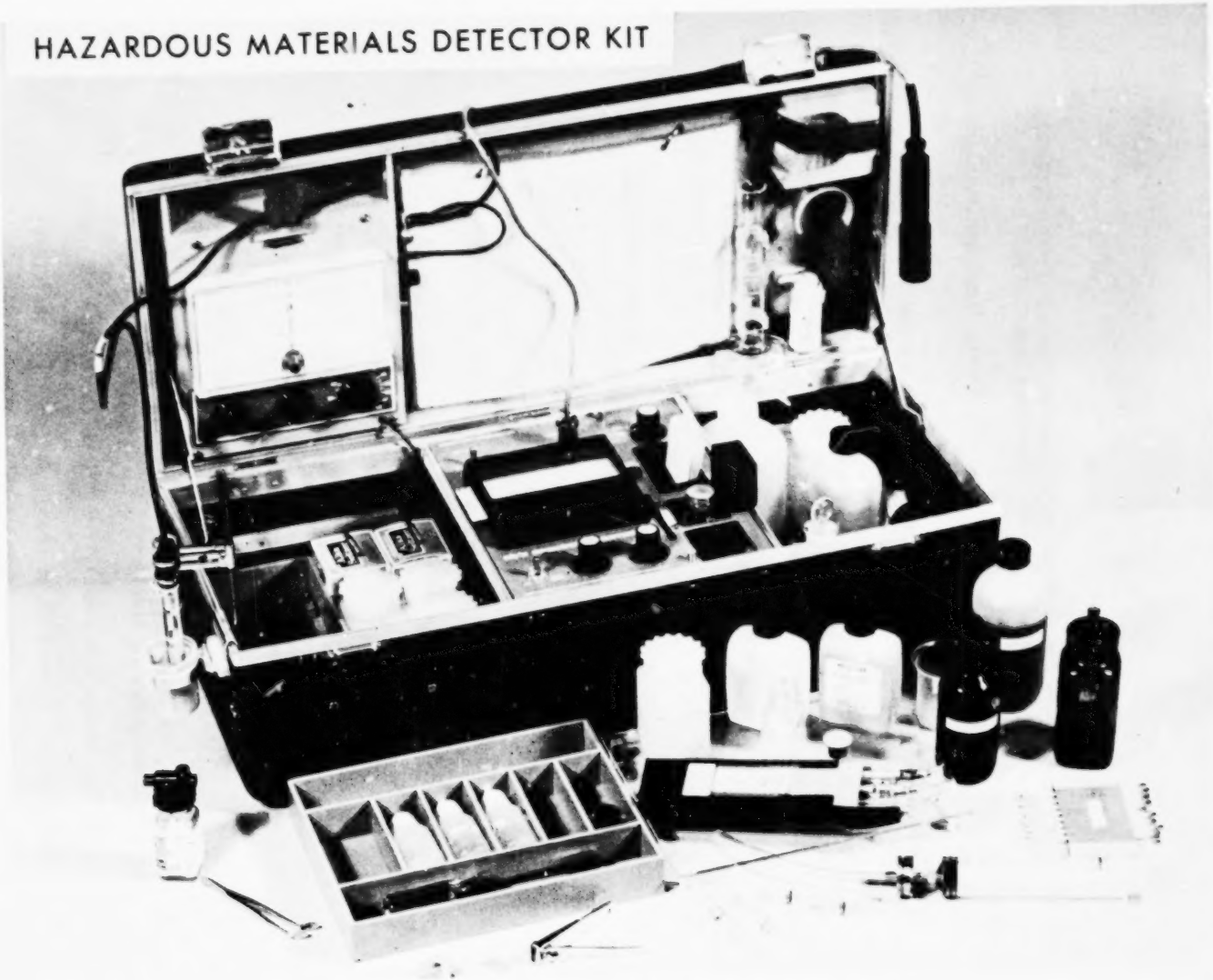

Figure 2 Hazardous Materials Detector Kit, In Use

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LIST OF MANUFACTURERS AND SUPPLIERS

1. Anderson Laboratories Corporation, Fort Worth, TX
2. Bausch and Lomb, Analytical Systems Division, Rochester, NY
3. Bendix/Gastec (Products available from National Environmental Instruments, Inc., Warwick, RI)
4. Boehringer Mannheim GMBH, Biochemcial Department (Products available from Fisher Scientific Co.)
5. Chemtrix, Inc., Hillsboro, OR
6. Corning Glass Co. (Electrodes are available from VWR Scientific Division of Univar)
7. Hach Chemical Co., Ames IA
8. International Ecological Systems Corporation, Mt. Laurel, NJ
9. Kitagawa (Products are available from Matheson Gas Products, East Rutherford, NJ)
10. Micro-Essential Laboratory, Brooklyn, NY
11. Rohm and Haas, Philadelphia, PA
12. Worthington Biochemical Corporation, Freehold, NJ

APPENDIX A
PROBABLE RESPONSES OF DETECTOR KIT TO MATERIALS LISTED IN FEDERAL REGISTER, VOL. 40, NO. 250, DEC. 75

	Chol. Inhib.	Benzene	Heavy Metals	Phenol	Cyanide	pH	Conductivity	Nitrate N_2	Color	Sulfate	Phosphate	Ammonia N_2	Chloride	Fluoride			
Acetaldehyde																	
Acetic acid						+											
Acetic anhydride						+											
Acetone cyanohydrin					+			+					+				
Acetyl bromide													+				
Acetyl chloride													+				
Acrolein																	
Acrylonitrile																	
Adiponitrile																	
Aldrin																	
Allyl alcohol																	
Allyl chloride																	
Aluminum sulfate						+	+			+				+			
Ammonia						+	+					+					
Ammonium Compounds:																	
Ammonium acetate							+					+					
Ammonium benzoate							+					+					
Ammonium bicarbonate							+					+					
Ammonium bisulfite							+					+					
Ammonium bromide							+					+	+				
Ammonium carbamate							+					+					
Ammonium carbonate							+					+					
Ammonium chloride							+					+	+				
Ammonium citrate, dibasic							+					+					
Ammonium ferrocyanide					+		+					+	+				
Ammonium fluoborate							+					+					
Ammonium formate							+					+					
Ammonium gluconate							+					+					
Ammonium hydroxide						+	+					+					
Ammonium hypophosphite												+					

APPENDIX A
PROBABLE RESPONSES OF DETECTOR KIT TO MATERIALS LISTED IN FEDERAL REGISTER, VOL. 40, NO. 250, DEC. 75 (continued)

	Chol. Inhib.	Benzene	Heavy Metals	Phenol	Cyanide	pH	Conductivity	Nitrate N_2	Color	Sulfate	Phosphate	Ammonia N_2	Chloride	Fluoride				
Ammonium iodide							+					+	+					
Ammonium molybdate							+	+				+						
Ammonium nitrate							+					+						
Ammonium oxalate							+					+						
Ammonium pentaborate							+					+		+				
Ammonium silicofluoride							+					+						
Ammonium sulfamate							+			+		+						
Ammonium sulfate							+					+	+					
Ammonium sulfide							+			+		+						
Ammonium sulfite							+					+						
Ammonium tartrate							+					+						
Ammonium thiocyanate							+			+		+						
Ammonium thisulfate																		
Amyl acetate																		
Aniline																		
Antimony Compounds:																		
Antimony pentachloride							+						+					
Antimony pentafluoride							+							+				
Antimony potassium tartrate							+						+					
Antimony tribromide							+						+					
Antimony trichloride							+							+				
Antimony trifluoride							+		+				+					
Antimony triiodide																		
Antimony trioxide																		
Arsenic Compounds:																		
Arsenic acid						+	+											
Arsenic disulfide																		
Arsenic pentaoxide						+	+											

APPENDIX A
PROBABLE RESPONSES OF DETECTOR KIT TO MATERIALS LISTED IN FEDERAL REGISTER, VOL. 40, NO. 250, DEC. 75 (continued)

	Chol. Inhib.	Benzene	Heavy Metals	Phenol	Cyanide	pH	Conductivity	Nitrate N_2	Color	Sulfate	Phosphate	Ammonia N_2	Chloride	Fluoride			
Arsenic tribromide							+						+				
Arsenic trichloride							+						+				
Arsenic trifluoride							+							+			
Arsenic triiodide							+						+				
Arsenic trioxide							+										
Arsenic trisulfide																	
Cacodylic acid																	
Calcium arsenate											+						
Calcium arsenite											+						
Potassium arsenate							+				+						
Potassium arsenite							+				+						
Sodium arsenate							+				+						
Sodium arsenite							+				+						
Sodium cacodylate							+										
Benzene		+															
Benzoic acid						+											
Benzonitrile																	
Benzoyl chloride													+				
Benzyl chloride		+															
Beryllium Compounds:																	
Beryllium chloride							+						+				
Beryllium fluoride							+							+			
Beryllium hydroxide						+											
Beryllium nitrate							+	+									
Beryllium phosphate							+				+						
Beryllium sulfate							+			+							
Boric acid						+											
Brucine																	
Butyl acetate																	

APPENDIX A
PROBABLE RESPONSES OF DETECTOR KIT TO MATERIALS LISTED IN FEDERAL REGISTER, VOL. 40, NO. 250, DEC. 75 (continued)

	Chol. Inhib.	Benzene	Heavy Metals	Phenol	Cyanide	pH	Conductivity	Nitrate N_2	Color	Sulfate	Phosphate	Ammonia N_2	Chloride	Fluoride			
Butylamine												+					
Butyric acid						+											
Cadmium Compounds:																	
Cadmium acetate			+				+										
Cadmium bromide			+				+						+				
Cadmium chloride			+				+						+				
Cadmium fluoborate			+				+										
Cadmium nitrate			+				+										
Cadmium sulfate			+				+			+							
Calcium carbide						+											
Calcium hydroxide						+											
Calcium hypochlorite			+			+	+					+	+				
Calcium oxide						+											
Captan																	
Carbaryl	+			+								+	+				
Carbon disulfide		+															
Catechol				+													
Chlordane																	
Chlorine			+			+	+						+				
Chlorobenzene		+															
Chloroform																	
Chlorosulfonic acid	+					+	+			+			+	+			
Chromium Compounds:																	
Ammonium bichromate							+		+			+					
Ammonium chromate							+		+			+					
Calcium chromate																	
Chromic acid						+	+		+								
Chromic sulfate						+	+		+	+							

APPENDIX A
PROBABLE RESPONSES OF DETECTOR KIT TO MATERIALS LISTED IN FEDERAL REGISTER, VOL. 40, NO. 250, DEC. 75 (continued)

	Chol. Inhib.	Benzene	Heavy Metals	Phenol	Cyanide	pH	Conductivity	Nitrate N_2	Color	Sulfate	Phosphate	Ammonia N_2	Chloride	Fluoride			
Chromous carbonate							+		+								
Chromous chloride							+		+				+				
Chromous oxalate							+		+								
Chromyl chloride						+	+		+				+				
Lithium bichromate							+		+								
Lithium chromate							+		+								
Potassium bichromate							+		+								
Potassium chromate							+		+								
Sodium bichromate							+		+								
Sodium chromate							+		+								
Strontium chromate																	
Zinc bichromate			+				+		+								
Cobalt Compounds:																	
Cobaltous acetate			+			+			+								
Cobaltous bromide			+				+		+				+				
Cobaltous chloride			+				+		+				+				
Cobaltous citrate			+				+		+								
Cobaltous fluoride			+				+		+					+			
Cobaltous formate			+				+		+								
Cobaltous iodide			+				+		+				+				
Cobaltous nitrate			+				+	+	+								
Cobaltous perchlorate			+				+		+								
Cobaltous succinate			+														
Cobaltous sulfamate			+														
Cobaltous sulfate			+				+		+	+							
Copper Compounds:																	
Cupric acetate			+				+		+								
Cupric acetoarsenite			+						+								

APPENDIX A

PROBABLE RESPONSES OF DETECTOR KIT TO MATERIALS LISTED IN FEDERAL REGISTER, VOL. 40, NO. 250, DEC. 75 (continued)

	Chol. Inhib.	Benzene	Heavy Metals	Phenol	Cyanide	pH	Conductivity	Nitrate N_2	Color	Sulfate	Phosphate	Ammonia N_2	Chloride	Fluoride			
Cupric acetylacetonate			+				+										
Cupric bromide			+				+		+				+				
Cupric chloride			+				+		+				+				
Cupric formate			+			+	+		+								
Cupric gluconate			+				+		+								
Cupric glycinate			+				+										
Cupric lactate			+				+										
Cupric nitrate			+				+	+									
Cupric oxalate			+				+										
Cupric subacetate			+				+		+								
Cupric sulfate			+				+		+	+							
Cupric sulfate, ammoniated			+				+		+			+					
Cupric tartrate			+				+		+								
Cuprous bromide			+				+						+				
Cuprous iodide			+				+		+				+				
Coumaphos	+																
Cresol				+													
Cyanide Compounds:																	
Barium cyanide					+		+						+				
Calcium cyanide					+		+						+				
Hydrogen cyanide					+		+						+				
Potassium cyanide					+		+						+				
Sodium cyanide					+		+						+				
Zinc cyanide			+		+		+						+				
Cyanogen chloride					+		+						+				
Cyclohexane					+								+				
2,4-D (acid)		+				+											
2,4-D (esters)																	
Dalapon						+											

APPENDIX A

PROBABLE RESPONSES OF DETECTOR KIT TO MATERIALS LISTED IN FEDERAL REGISTER, VOL. 40, NO. 250, DEC. 75 (continued)

	Chol. Inhib.	Benzene	Heavy Metals	Phenol	Cyanide	pH	Conductivity	Nitrate N_2	Color	Sulfate	Phosphate	Ammonia N_2	Chloride	Fluoride			
DDT																	
Diazinon	+																
Dicamba																	
Dichlobenil		+															
Dichlone																	
Dichlorvos	+																
Dieldrin												+					
Diethylamine												+					
Dimethylamine												+					
Dinitrobenzene									+								
Dinitrophenol				+					+								
Diquat							+						+				
Disulfoton	+																
Diuron																	
Dodecyl benzenesulfonic acid						+											
Dodecyl benzenesulfonic acid, calcium salt							+										
Dodecyl benzenesulfonic acid isopropanol amine salt												+					
Dodecyl benzenesulfonic acid, sodium salt						+	+										
Dodecyl benzenesulfonic acid, triethanolamine salt																	
Dursban	+																
Endosulfan																	
Endrin																	
Ethion	+																
Ethylbenzene		+															
Ethylenediamine												+					
Ethylenediamine-tetraacetic acid												+					

APPENDIX A

PROBABLE RESPONSES OF DETECTOR KIT TO MATERIALS LISTED IN FEDERAL REGISTER, VOL. 40, NO. 250, DEC. 75 (continued)

	Chol. Inhib.	Benzene	Heavy Metals	Phenol	Cyanide	pH	Conductivity	Nitrate N_2	Color	Sulfate	Phosphate	Ammonia N_2	Chloride	Fluoride			
Fluorine Compounds:																	
Aluminum fluoride						+											
Ammonium bifluoride						+	+					+		+			
Ammonium fluoride						+	+					+		+			
Hydrofluoric acid						+	+							+			
Lithium fluoride						+	+							+			
Phosphorus pentafluoride						+	+				+			+			
Sodium bifluoride						+	+							+			
Sodium fluoride						+	+							+			
Stannous fluoride						+	+							+			
Formaldehyde						+											
Formic acid						+											
Fumaric acid																	
Furfural																	
Guthion	+											+					
Heptachlor						+	+							+			
Hydrochloric acid				+													
Hydroquinone												+					
Hydroxylamine																	
Iron Compounds:																	
Ferric ammonium citrate							+					+					
Ferric ammonium oxalate							+					+					
Ferric chloride							+						+				
Ferric fluoride														+			
Ferric glycerophosphate							+										
Ferric nitrate							+	+									
Ferric phosphate											+						
Ferric sulfate										+							

APPENDIX A
PROBABLE RESPONSES OF DETECTOR KIT TO MATERIALS LISTED IN FEDERAL REGISTER, VOL. 40, NO. 250, DEC. 75 (continued)

	Chol. Inhib.	Benzene	Heavy Metals	Phenol	Cyanide	pH	Conductivity	Nitrate N_2	Color	Sulfate	Phosphate	Ammonia N_2	Chloride	Fluoride			
Ferrous ammonium sulfate			+				+			+		+					
Ferrous chloride			+				+						+				
Ferrous oxalate			+														
Ferrous sulfate			+							+							
Isoprene		+															
Kelthane																	
Lead Compounds:																	
Lead acetate			+				+										
Lead arsenate			+								+						
Lead bromide			+										+				
Lead chloride			+										+				
Lead fluoborate			+														
Lead fluoride			+											+			
Lead iodide			+										+				
Lead nitrate			+				+	+									
Lead stearate			+														
Lead sulfate			+							+							
Lead sulfide			+										+				
Lead tetraacetate			+														
Lead thiocyanate			+														
Lead thiosulfate			+														
Lead tungstate			+														
Lindane																	
Malathion	+	+				+						+					
Maleic acid						+											
Maleic anhydride						+											

APPENDIX A
PROBABLE RESPONSES OF DETECTOR KIT TO MATERIALS LISTED IN FEDERAL REGISTER, VOL. 40, NO. 250, DEC. 75 (continued)

	Chol. Inhib.	Benzene	Heavy Metals	Phenol	Cyanide	pH	Conductivity	Nitrate N_2	Color	Sulfate	Phosphate	Ammonia N_2	Chloride	Fluoride				
Mercury Compounds:																		
Mercuric acetate			+				+											
Mercuric ammonium chloride			+				+					+	+					
Mercuric bromide			+										+					
Mercuric chloride			+				+						+					
Mercuric cyanide			+		+		+						+					
Mercuric iodide			+															
Mercuric nitrate			+					+										
Mercuric oxide			+															
Mercuric sulfate			+							+								
Mercuric thiocyanate			+															
Mercurous chloride			+															
Mercurous iodide			+										+					
Mercurous nitrate			+					+										
Methoxychlor																		
Methyl mercaptan		+																
Methyl methacrylate																		
Methyl parathion	+																	
Mevinphos	+																	
Molybdic trioxide																		
Monoethylamine																		
Naled	+											+						
Naphthalene		+																
Naphthalenic acid																		
Nickel Compounds:																		
Nickel acetate			+				+		+									
Nickel ammonium sulfate			+				+		+	+		+						
Nickel bromide			+				+						+					

APPENDIX A

PROBABLE RESPONSES OF DETECTOR KIT TO MATERIALS LISTED IN FEDERAL REGISTER, VOL. 40, NO. 250, DEC. 75 (continued)

	Chol. Inhib.	Benzene	Heavy Metals	Phenol	Cyanide	pH	Conductivity	Nitrate N_2	Color	Sulfate	Phosphate	Ammonia N_2	Chloride	Fluoride			
Nickel chloride			+				+						+				
Nickel fluoride			+				+		+					+			
Nickel formate			+				+		+								
Nickel hydroxide			+						+								
Nickel iodide			+				+						+				
Nickel nitrate			+				+	+	+								
Nickel perchlorate			+				+										
Nickel sulfate			+				+			+							
Nitric acid						+	+	+									
Nitrobenzene		+															
Nitrogen dioxide						+		+									
Nitrophenol						+			+			+		+			
Paraformaldehyde																	
Parathion	+								+			+					
Pentachlorophenol				+													
Phenol				+													
Phosgene						+	+						+				
Phosphoric acid						+	+				+						
Phosphorus						+	+				+						
Phosphorus oxychloride						+	+				+		+				
Phosphorus pentasulfide		+				+	+	+			+	+	+	+			
Phosphorus trichloride						+	+				+		+				
Polychlorinated biphenyls																	
Potassium hydroxide						+	+										
Potassium permanganate							+		+								
Propionic acid						+											
Propionic anhydride						+											
Propyl alcohol																	
Pyrethrins																	
Pyrogallic acid				+													
Quinoline		+															
Resorcinol				+													

APPENDIX A

PROBABLE RESPONSES OF DETECTOR KIT TO MATERIALS LISTED IN FEDERAL REGISTER, VOL. 40, NO. 250, DEC. 75 (continued)

	Chol. Inhib.	Benzene	Heavy Metals	Phenol	Cyanide	pH	Conductivity	Nitrate N_2	Color	Sulfate	Phosphate	Ammonia N_2	Chloride	Flouride			
Selenium Compounds:							+										
Selenic acid							+						+				
Selenium oxide							+										
Selenium oxychloride							+										
Sodium selenite						+	+										
Sodium						+	+										
Sodium bisulfite							+						+				
Sodium borate							+										
Sodium hydrosulfide						+	+						+				
Sodium hydroxide			+			+	+										
Sodium hypochlorite						+	+	+			+						
Sodium methylate							+				+						
Sodium nitrite						+	+				+						
Sodium phosphate, dibasic						+	+				+						
Sodium phosphate, monobasic							+						+				
Sodium phosphate, tribasic		+					+										
Sodium silicate																	
Sodium sulfide		+															
Strychnine						+	+			+							
Styrene						+	+						+				
Sulfuric acid		+				+	+										
Sulfur monochloride		+				+											
2,4,5-T (acid)																	
2,4,5-T (esters)						+											
Tannic acid																	
TDE																	
Tetraethyl lead	+																
Tetraethyl pyrophosphate		+															
Toluene																	

APPENDIX A

PROBABLE RESPONSES OF DETECTOR KIT TO MATERIALS LISTED IN FEDERAL REGISTER, VOL. 40, NO. 250, DEC. 75 (continued)

	Chol. Inhib.	Benzene	Heavy Metals	Phenol	Cyanide	pH	Conductivity	Nitrate N_2	Color	Sulfate	Phosphate	Ammonia N_2	Chloride	Fluoride
Toxaphene														
Trichlorfon	+													
Trichlorophenol				+										
Triethylamine														
Trimethylamine														
Uranium Compounds:														
Uranium peroxide							+							
Uranyl acetate							+	+						
Uranyl nitrate							+			+				
Uranyl sulfate														
Vanadium Compounds:														
Vanadium oxytrichloride							+						+	
Vanadium pentoxide														
Vanadyl sulfate							+			+				
Vinyl acetate														
Xylenol				+										
Xylene		+												
Zectran														
Zinc Compounds:														
Zinc acetate			+				+					+	+	
Zinc ammonium chloride			+				+					+		
Zinc ammonium sulfate			+				+			+		+		
Zinc borate			+				+						+	
Zinc bromide			+				+							
Zinc carbonate			+										+	
Zinc chloride			+				+							+
Zinc fluoride			+				+							

APPENDIX A

PROBABLE RESPONSES OF DETECTOR KIT TO MATERIALS LISTED IN FEDERAL REGISTER, VOL. 40, NO. 250, DEC. 75 (continued)

	Chol. Inhib.	Benzene	Heavy Metals	Phenol	Cyanide	pH	Conductivity	Nitrate N_2	Color	Sulfate	Phosphate	Ammonia N_2	Chloride	Fluoride			
Zinc formate			+			+	+										
Zinc hydrosulfite			+				+										
Zinc nitrate			+			+	+	+									
Zinc permanganate			+				+		+								
Zinc phenolsulfonate			+	+			+										
Zinc phosphide			+														
Zinc potassium chromate			+				+		+								
Zinc propionate			+				+										
Zinc silicofluoride			+				+										
Zinc sulfate			+				+			+							
Zinc sulfate, monohydrate			+				+			+							
Zirconium Compounds:																	
Zirconium acetate							+										
Zirconium ammonium fluoride							+					+		+			
Zirconium potassium fluoride							+							+			
Zirconium nitrate							+	+									
Zirconium oxychloride							+						+				
Zirconium sulfate							+			+							
Zirconium tetrachloride							+						+				

APPENDIX B

OPERATOR'S MANUAL FOR HAZARDOUS MATERIALS DETECTOR KIT

SECTION I

INTRODUCTION

The Hazardous Materials Detector Kit is a portable self-sufficient laboratory capable of providing the investigator in the field with a variety of valuable information to assist him in detecting (not necessarily identifying) and tracing contamination in inland waters. While similar to other water test kits, the equipment and procedures contained are designed to be used as a part of a total concept. Before attempting to use the kit in an investigation, the operator should thoroughly familarize himself with the concept of use and obtain an understanding of the significant data that can be derived. Special apparatus, tests taken from commercial and military kits, and procedures designed specifically for this item form the makeup of the kit.

The kit contains delicate instrumentation and should be treated accordingly. Care and maintenance suggested in the manuals should serve to prolong its usefulness. Instrumentation consists of a battery or line-operated spectrophotometer and a conductivity meter contained in a Hach Chemical Co. DR-EL/2 Laboratory enclosure. In addition, the Hach Model 1975 battery-operated pH meter has been modified and incorporated in the enclosure. Hach manuals for the DR-EL/2 Laboratory and Model 1975 pH Meter are provided with the kit and should be referred to frequently for operation, service and maintenance. In addition to this manual and the Hach manuals, abbreviated, laminated instruction cards (sufficient to provide the operator in the field with guidance) are included. Data sheets are provided for reporting and recording data.

To assist the operator in quickly gaining familiarity with use of the kit, this manual is divided into sections: (a) Concept of Use, (b) Equipment and Methodology, (c) Analytical Procedures and (d) Consumable Materials.

SECTION II

CONCEPT OF USE

Originally, it was intended to develop a field kit using a standardized system, in which results are easily obtained and interpreted by a senior technician. These goals, in part, have been met; the tests are easy to perform, short, and designed for the greatest ease of operation in the field. However, the task of detecting almost any contaminant in any water background is a formidable one and requires the ingenuity and interpretive skills of a dedicated investigater. The kit combines instrumental and wet chemical methods; in the hands of a chemist with experience in water chemisty, it can provide the data necessary to detect and follow contamination in inland waterways. Use of the kit relies basically on three types of evaluations as shown in the table below. Details of these procedures are given in the section on Analytical Procedures.

Detection Procedures of the Hazardous Materials Detector Kit

General Assessment	Relative Measurements
a. Appearance	i. pH
b. Color of Filtered Water	j. Conductivity
c. Color of Suspended Matter	k. Turbidity
Spot Tests	l. Nitrate Nitrogen
d. Cholinesterase Inhibitors	m. Color
e. Benzene	n. Sulfate
f. Heavy Metals	o. Phosphate
g. Phenol	p. Ammonia Nitrogen
h. Cyanide	q. Chloride
	r. Fluoride

The tests as a whole are largely nonspecific and are capable of detecting a much wider range of contaminants than is indicated. General

Assessment procedures (a to c) allow the operator to make a general evaluation of the water to be tested. Spot Tests procedures (d to h) indicate that a particular type of contaminant is or is not present. However, any positive response by the Spot Tests should be considered as contamination since this indicates a property not normally found or desired in water. The final group of tests (i to r), Relative Measurements, is based on a natural property of water. Contamination is indicated when a significant increase in one of these parameters is observed over uncontaminated water.

While use of the kit cannot be standardized for all situations, a systematic approach can make gathering of data more direct and meaningful. First the investigator assesses the presence of contamination by evaluating two major factors: (a) The nature of the water source -- is it stationary, like a lake or a pond, or is it moving like a stream or river; and (b) The nature of the contaminant -- is it specifically known, totally unknown or partially known, such that it may be acid or base, organic or inorganic, phosphate containing, colored, etc.

Second the investigator confronts the situation in an orderly fashion: (a) He immediatly conducts a "General Assessment" (tests a to c); (b) He considers using Spot Tests (4 to 8) since these are simple and easy to evaluate. If he knows, or suspects strongly, that the contaminant will respond to one of these tests, this may be the only tool he needs to conduct his investigation; and (c) He goes to Relative Measurements (tests 9 to 18). If location of the contaminant is based on a relative measurement then he must make a comparison between the area of suspected contamination and a clean source of water (reference sample). In the case of a stream, the reference sample may be taken at a location upstream from the contamination. If the body of water is a lake, he must use his best judgment and select an area believed to be clean. Once the reference water is established, it is a matter of determining which relative tests will be most useful in detecting significant differences.

When the pollutant is totally unknown, the investigator may have to run the entire series of tests, looking for those revealing the most significant differences. These differences may then be taken as indicators of suspected contamination. Thereafter, only tests showing the significant differences need be run on suspect samples.

In those cases where some information is known about the contaminant, it may be sufficient to run only tests which are relevant. For example, if the investigator knows the contaminant to be an inorganic acid, he may proceed immediately to pH and conductivity measurements and run tests for inorganic ions. For example, the investigator knows that the contaminant is chlorosulfonic acid. He has options to make pH, conductivity, chloride and/or sulfate determinations. He would probably select several parameters which have the lowest background reading with respect to the reference water.

If the investigator knows the contaminant to be detected but not if it responds to any of the tests, he may wish to make some quick spot tests in the laboratory before taking the kit into the field.

After the investigator has selected indicators for contamination he will want to record his data carefully and systematically. The gradual change in the data will eventually provide clues to the location and level of contamination. Data sheets designed specifically for this purpose are included in the kit.

As indicated earlier, use of the kit cannot be fully standardized; the investigator must be ready at each occasion to make judgments relative to the surroundings and the circumstances of the contamination. In the procedures section all possible anticipated reactions to the tests have been noted. If, however, in the performance of a test, a precipitate or unusual color not indicated occurs in the test sample but not the reference sample, then it is very possible that contamination of some sort is present.

In making determinations with the spectrophotometer, it may often suffice simply to mark the test positive when a significant color indicative of contamination starts to develop. It may be adequate for the investigator to know the sample is still heavily contaminated without actually determining a specific value.

Perhaps the most difficult judgment to make is to determine how great a difference between the test sample and reference water sample is significant in the Relative Measurements tests. It will take familiarization with the kit and experience in its application to make reliable assessments in a given situation. As a "rule-of-thumb", based on the studies conducted to date, the following are considered significant instrumental changes:

(a) Change in pH of 0.5 units.

(b) 20 percent gain in conductivity.

(c) 10 percent gain in the full scale deflection (not concentration) in spectrophotometric procedures.

Experience may show that these values are conservative and smaller differences will be found significant.

As a final note, a fluorescent dye marker is included in the kit. This may be of use in marking the location of a contaminant.

SECTION III

EQUIPMENT AND METHODOLOGY

General. With the exception of a container to collect water samples, the Hazardous Materials Detector Kit contains the necessary instrumentation, equipment and reagents to perform all the methods described in Analytical Procedures. This manual provides the user with an overview sufficient for operation of the instruments. Instruments are from the Hach Chemical Company and Hach manuals are provided as part of the detector kit package. Hach manuals should be consulted for operation, maintenance, and servicing of their instruments.

Power. The instruments are powered by batteries for field use. In addition, the spectrophotometer and conductivity meter may be modified for 120 or 240 VAC operation using an adapter and cable provided with the kit. The procedure for the conversion is described in the Hach manual. The pH meter will operate only from the battery supply.

Batteries required for the kit are 1 each, 6.75 V, Mallory TR-235R (or equivalent), 8 each, 1.5 V size D, and 4 each 1.5 V size AA. It is recommended that expendable alkaline manganese batteries be used, when available, for economy and widest environmental range of operation. The investigator should minimize the on-time of all instrumentation in order to conserve battery life in the field.

Batteries may be checked by the following procedure:

(a) Set the POWER switch on the pH meter to the BATT CHECK position. If the needle deflects beyond BAT OK, the battery is alright. Otherwise replace the 6.75 V battery.

(b) Set the conductivity meter range switch to the BATT CHECK position. Hold the BATT CHECK switch on the spectrophotometer face in the DR position to check only the D size batteries. The needle should deflect beyond 83 percent. Hold the same BATT CHECK switch in the COND position to check both the D and AA size batteries. Again the needle should deflect 83 percent.

Batteries may be reached for servicing by unloosening the two knurled screws and removing the spectrophotometer. Arrangement of the batteries is shown in Figure 1.

Loading Diagram. Location of the kit components is shown in Figure 2. The tray is easily removed by lifting the top out by one of the finger holes and then grasping the inner partitions of the tray and lifting straight up.

Setting Up the Kit in the Field. The investigator should attempt to find several square feet of flat area. A raised surface like a mound or large rock would give greater operating comfort. The investigator may use a layout according to his own requirements, but that shown in Figure 3 is convenient for reaching all necessary pieces. Note that the tray top doubles as a small work surface. The arrangement of the pH measuring components is shown in Figure 4.

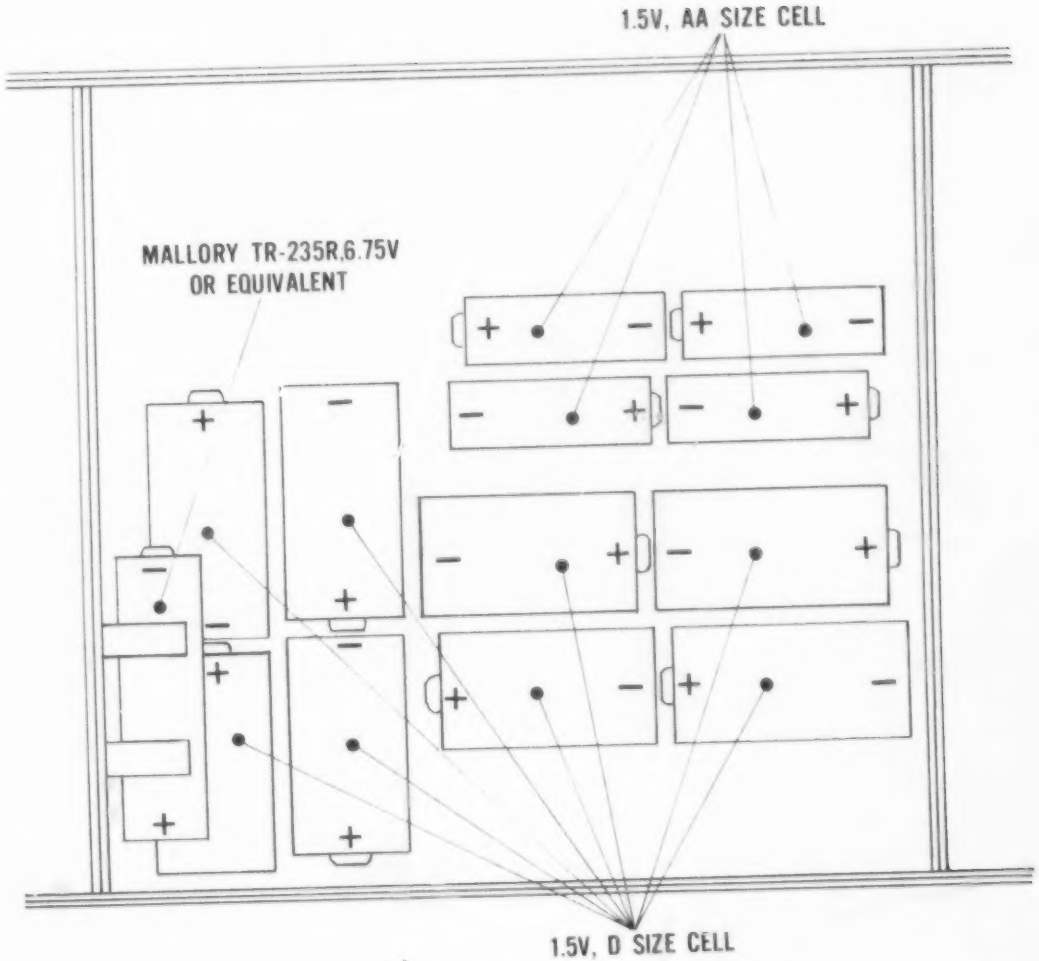

Figure 1. BATTERY ARRANGEMENT

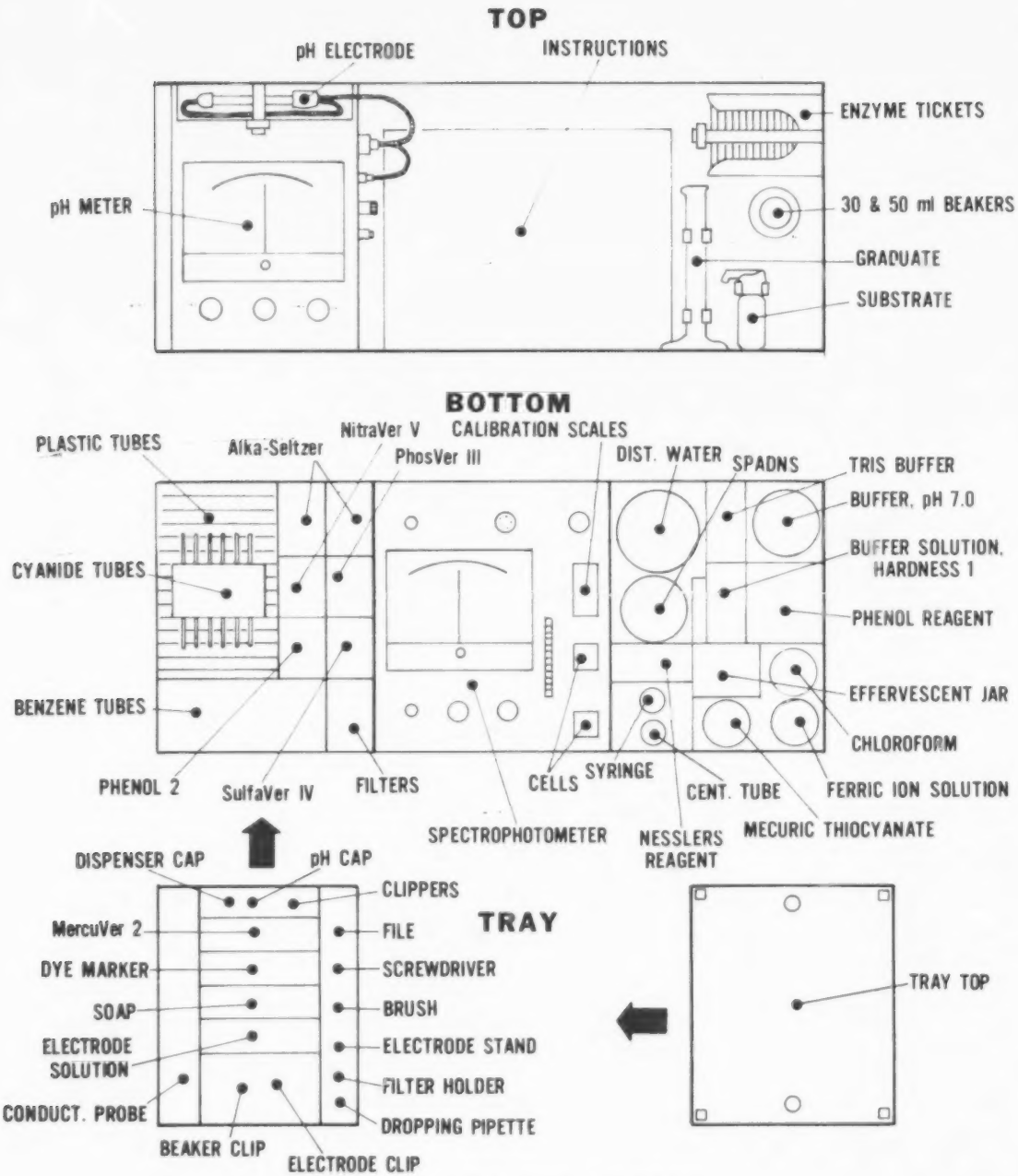

Figure 2. LOADING DIAGRAM

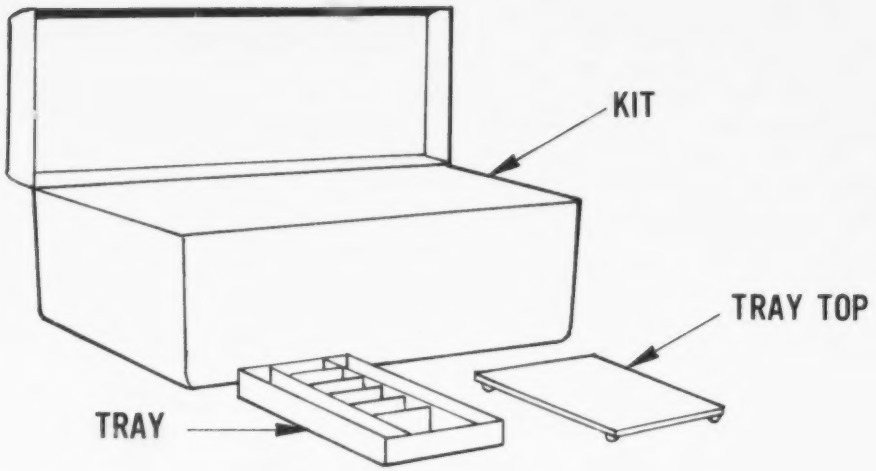

Figure 3. TYPICAL FIELD SETUP

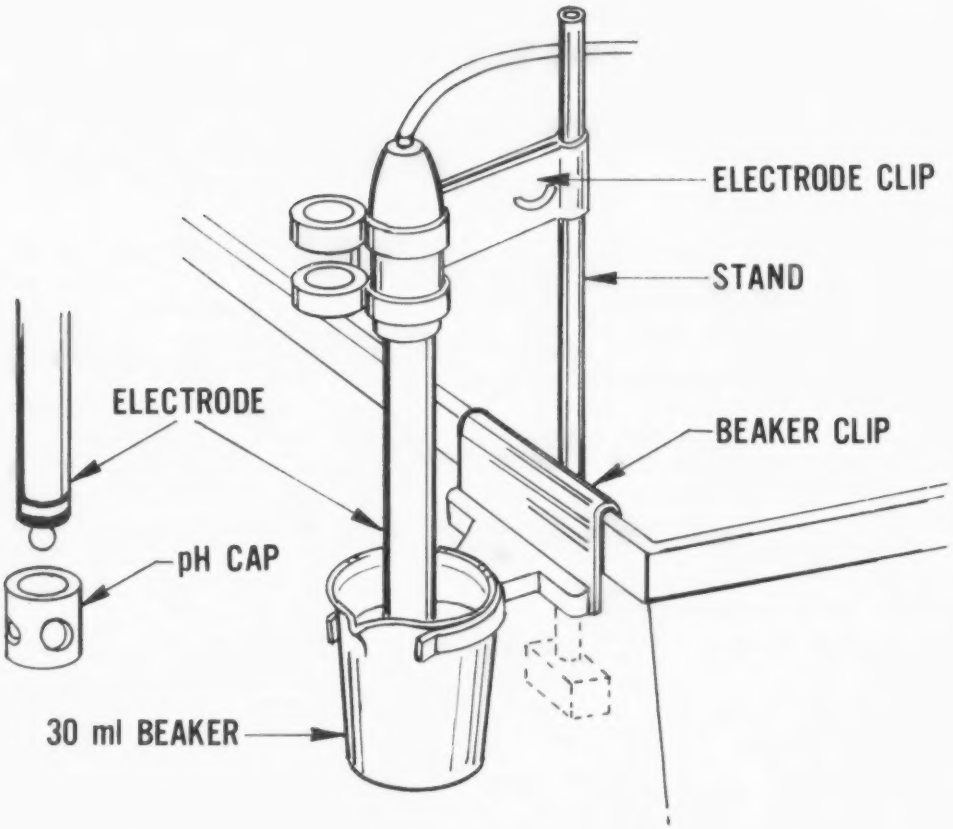

Figure 4. pH MEASURING COMPONENTS

pH Meter. Hang the 30 ml beaker on the edge of the case using the beaker clip as shown in Figure 4. Stand the support rod, with the electrode spring clip in place, in the vertical hole located inside the left front corner of the case. Remove the wetting cap from the electrode and replace with the plastic bulb protector. Install the electrode in the spring clip and set at a suitable height to reach into the beaker. Fill the beaker with distilled water to start the electrode soaking immediately. The liquid junction must always be below the water level. After about 10 minutes, fill the beaker with 25 ml of pH 7.0 buffer and immerse the electrode again. Set the TEMP dial to the approximate solution temperature and turn the POWER switch to the ON position. Adjust the STANDARDIZE switch to read exactly 7.0 and turn the power off. Discard the buffer, rinse the electrode and soak in distilled water. The instrument is ready for use.

The above procedure performed daily is adequate for field use. For more exacting standardization on a less frequent basis refer to the Hach manual.

Conductivity Meter. The conductivity probe requires no conditioning and is readied for use by plugging into the PROBE INPUT socket and inserting the Conductivity Meter Scale into the meter. The probe is submersed in water sample sufficient to cover the vent holes. The RANGE switch is placed in the "5" position and is worked down until an on-scale reading is obtained. If the reading is in the lower 10 percent of the scale, the next lower RANGE position should be used.

Spectrophotometer. The Hach Spectrophotometer is designed to be used with insertable meter scales which are provided. (If the operator wishes to devise his own analytical procedures, he may prepare calibration scales following instructions in the Hach manual and using blank meter scales available for this purpose.)

The spectrophotometer should be adjusted to zero before each use, but need not be done so before each test. Using a right-to-left reading meter scale, hold the light switch in the ZERO CHECK position and use the ZERO ADJUST knob to set the meter needle to the extreme left mark of the scale.

Each time the spectrophotometer is used the proper meter scale is inserted and the wavelength is set. A sample cell containing a blank prepared as described in the procedures is inserted and, after turning the instrument on, the LIGHT CONTROL switch is adjusted to zero on the meter scale. Once the instrument is zeroed, a cell containing the true sample is placed in the spectrophotometer. The instrument is turned on and the meter reading is noted. All readings with the instrument should be sufficiently long to obtain a stable reading, still the on-time should be kept to a minimum to prolong battery life.

In those instances where the concentration of a component is so high as to run off scale, a dilution should be made. The 1 ml dropping pipet and the 25 ml graduate may be used for this purpose. The scale reading will then be multiplied by the dilution factor.

Filter Assembly. In making spectrophotometric determinations with the exception of turbidity measurement, filtered water samples are used. Because gravity filtration is too slow, the assembly shown in Figure 5, for which glass fiber filters are provided, is used to quickly obtain clear samples. Filter changes are easily made using the flat ended tweezers.

It may be desirable in some instances to save the filter discs in order to use the color accummulated on them as a means to monitor contamination. In this use, of course, each disc would be used only once and with a specified volume of water.

It is important that samples be filtered only when indicated. Many contaminants may be in the form of slicks, solids or emulsions and their removal prior to analysis in procedures such as the spot test may grossly affect the result of the test.

Effervescent Jar. The Effervescent Jar assembly (Figure 6) is used to detect contaminants in water which may be expelled by an effervescent material. Alka-Seltzer® tablets are used for effervescence and Bendix-Gastec benzene analyzer tubes, which are sensitive to a variety of contaminants are used as the detector.

The jar is filled to the level mark with the water sample (about 80 ml). The triangular file is used to score and remove the tips of an analyzer tube. The tube is then inserted in the cap assembly with arrow pointing away from the jar. An Alka-Seltzer® tablet is dropped into the jar and the cap quickly and snugly screwed on. The test is allowed to continue until effervescence ceases.

Caution should be taken not to drive water droplets into the analyzer tube since this would inhibit the reaction.

Chromatographic Tube Test. Kitagawa detector tubes included in the kit are miniature chromatographic columns impregnated with reagents to detect cyanide in water. The triangular file is used to score and remove the tips of the tube. The tube is stood (with the red ball pointed up) in a depth of 1-2 cm of water sample (Figure 7). As the water is drawn up the tube by capillary action, a blue stain will form on the silica gel if cyanide is present.

Enzyme Ticket. The enzyme ticket (Figure 8) contains glass fiber spots impregnated with horse serum cholinesterase. When wetted with pH 8.0 Tris buffer, or neutral water not containing a cholinesterase inhibitor, the enzyme becomes active and will change the color of the orange color substrate to blue. If the enzyme is exposed to a cholinesterase inhibitor such as certain insecticides, rodenticides or some other toxic materials, it is inactivated and unable to change the color of the substrate. One end of the ticket is immersed in the water sample and the other is used as a control.

Data Sheets. Forms shown in Figure 9 are provided with the kit and may be used for recording and reporting data.

General Maintenance. For specific maintenance and servicing problems regarding instrumentation refer to the Hach manuals. As a matter of general upkeep, some common sense approaches to the equipment will be helpful. Obviously, delicate instrumentation requires gentle handling; drops and severe jarring should be avoided. It may be desirable to pass a wire loop around the battery clips to prevent the cells from "popping" out.

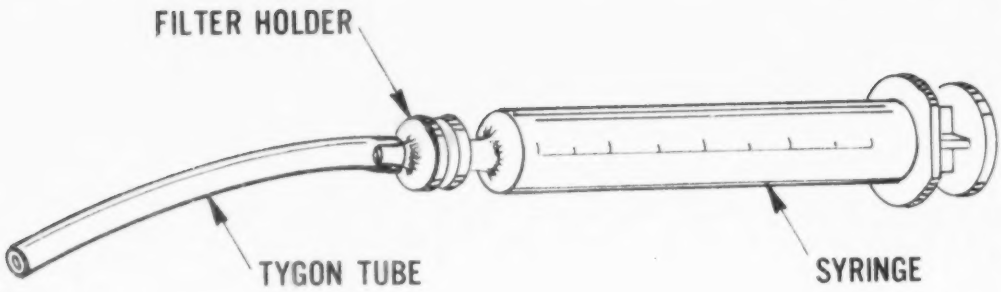

Figure 5. FILTER ASSEMBLY

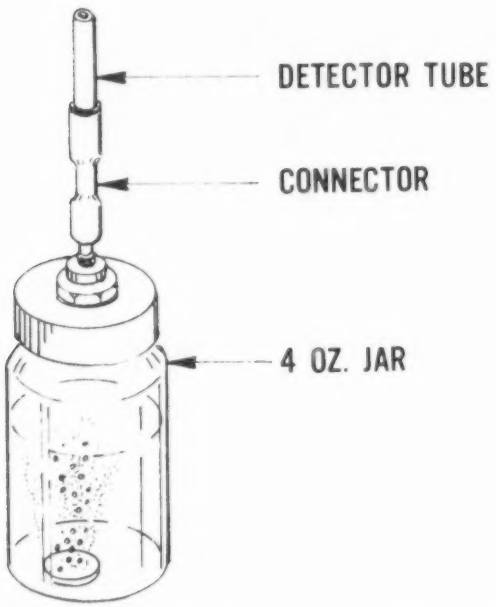

Figure 6. EFFERVESCENT JAR ASSEMBLY

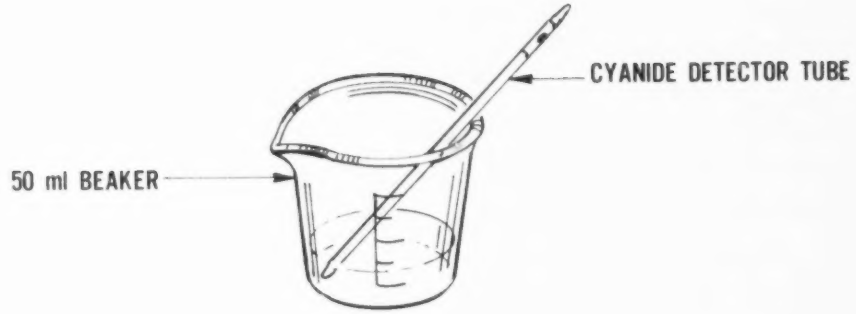

Figure 7. CHROMATOGRAPHIC TUBE TEST

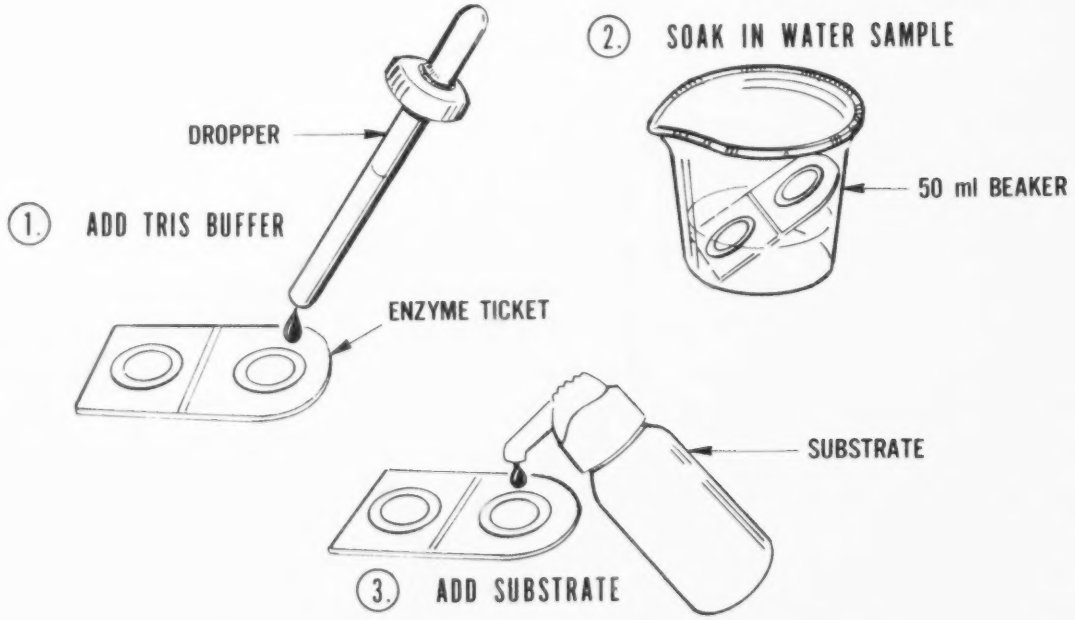

Figure 8. CHOLINESTERASE INHIBITOR TEST

FIGURE 9

DATA SHEET FOR HAZARDOUS MATERIALS DETECTOR KIT

GENERAL ASSESSMENT

SPOT TESTS

Sample	Time	Location	Appearance	Col. Water	Col. Matter	Chol. Inhib.	Benzene	H'vy Metals	Phenol	Cyanide
1										
2										
3										
4										
5										
6										
7										
8										
9										
10										

RELATIVE MEASUREMENT TESTS

Sample	pH	Conduct. umhos/cm.	Turbidity FTU	Nitrate, N_2 mg/l	Color APHA Units	Sulfate mg/l	Phosphate mg/l	Ammonia, N_2 mg/l	Chloride mg/l	Fluoride mg/l
1										
2										
3										
4										
5										
6										
7										
8										
9										
10										

The investigator should avoid getting excessive amounts of water into the kit. The electronics would be damaged; the filter wheel of the spectrophotometer is sensitive to extended periods of high humidity. Certainly, in returning equipment to the kit care should be taken to shake or wipe off excess water. On returning to the laboratory after a day in the field, allow the kit to stand open to dry out.

General cleaning procedures which may be applied to the conductivity probe, electrode, cells, photocell window and filter wedge are described in the Hach manuals.

Many of the specially fabricated plastic parts in the kit are made of Lucite® protected with a tough vinyl paint. While Lucite is quite strong, it may still be broken. In this event the broken pieces should be held in contact while a small amount of methylene chloride is made to flow into the crack with a small syringe. The pieces will bond immediately and in a few hours will be as strong as new.

Check List Before Going to the Field. The following should be used as a check list in the laboratory before taking the kit to the field.

(a) Check loading diagram to assure that all equipment and reagents are present. If more use of some reagents is anticipated, they should be brought in an additional container.

(b) Perform prescribed battery checks, and replace weak batteries where necessary.

(c) Check function (not standardize) of the pH meter, conductivity meter, and spectrophotometer.

SECTION IV

ANALYTICAL PROCEDURES

This section contains detailed procedures for the analysis of contaminants in water. The arrangement is made to correspond with the data sheet provided with the kit. Questions regarding equipment and its use should be referred to the previous section.

General Assessment

(a) APPEARANCE

Describe the water sample with respect to color, turbidity, suspended matter, slicks, etc.

(b) COLOR OF FILTERED WATER

Apparatus: Filter Assembly

Procedure: Filter 50 ml of the water sample. Record the visual color of the filtered water and save a sample in a plastic test tube for reference. Save the glass fiber filter for Procedure 3.

(c) COLOR OF SUSPENDED MATTER

Record the color of the matter collected on the glass fiber filter in the previous step. Save the filter for reference.

Spot Tests

(d) CHOLINESTERASE INHIBITORS

Reagents: Enzyme Tickets
Tris Buffer, pH 8.0
Substrate

Procedure:

1. Add about 20 ml of water sample to the 50 ml beaker.

2. Remove an enzyme ticket from the plastic sleeving. Add several drops of Tris Buffer to the test spot on the round end of the ticket and 1 ml to the beaker.

3. Stir the buffered water sample using the square end of the ticket; then allow the ticket to stand in the beaker (square end down), being careful not to wet the test spot on the round end (blank) with the water sample. Allow to stand for 5 minutes.

4. Remove the ticket from solution; shake off the excess water.

5. Add several drops of Substrate to each test spot and allow the blue color to develop.

6. At the end of about 2 minutes, the round end (blank) should be blue. Compare with the square end (test sample) of the ticket. If the square end is significantly different, i.e., no blue or only the very slightest blue, a contaminant, probably an anticholinesterase type, may be present.

(e) BENZENE

Apparatus: Effervescent Jar Assembly

Reagents: Alka-Seltzer® Tablets
Benzene Analyzer Tubes

Procedure:

1. Fill the Effervescent Jar to the level mark with the water sample.

2. Score the tips of a Benzene Analyzer Tube with the triangular file and remove.

3. Insert the tube into the tubing of the jar cap assembly with the arrow pointed away from the cap.

4. Add one Alka-Seltzer® tablet to the jar, screw the cap on immediately and allow the effervescence to go to completion.

5. Examine the analyzer tube for an orange, tan, brown, or greenish discoloration of the white silica gel. Since the tube is designed for air testing the concentration has no relation to water. However, the length of the stain may be recorded as a relative index of contamination.

(f) HEAVY METALS

Apparatus: Plastic Test Tube with Cap
Bottle Cap Dispenser

Reagents: Tris Buffer, pH 8.0
Chloroform
MercuVer™ 2 Powder

Procedure:

1. Add 2 ml of Tris Buffer, pH 8.0 to a new plastic test tube. Fill to the "WATER LEVEL" mark with the water sample; cap and shake to mix.

2. Add 1 ml of chloroform.

3. Assemble the MercuVer™ 2 reagent jar with the bottle cap dispenser;

add 1 shot to the test tube.

4. Shake for 30-40 seconds.

5. A negative test is indicated by the blue-green coloration of the lower chloroform layer. A significantly different color such as pink, deep purple, yellow or orange indicates a positive test for contamination. Any questionable samples should be compared with a distilled water blank.

(g) PHENOL

Apparatus: 40 ml Glass-stoppered Centrifuge Tube

Reagents: Buffer Solution, Hardness 1
Phenol Reagent Powder Pillows
Phenol 2 Powder Pillows
Chloroform

Procedure:

1. Fill the centrifuge tube to the 25 ml mark with the water sample.

2. Add 1 ml of Buffer Solution, Hardness 1 and mix.

3. Add the contents of a Phenol Reagent Powder Pillow; stopper and shake to dissolve. Ignore any precipitate which may form in the aqueous layer.

4. Add the contents of a Phenol 2 Powder Pillow; stopper and shake to dissolve.

5. Add 1 ml of chloroform and shake for 30-40 seconds.

6. An orangish to brown color or a rose to purple color in the chloroform layer indicates a positive test. A clear deep yellow color is negative. (If there is any doubt a blank may be run using distilled water.)

(h) CYANIDE

Reagents: Cyanide Ion Detector Tube

Procedure:

1. Fill the 50 ml beaker with 15-20 ml of water sample.

2. Score the tips of the Cyanide Ion Detector Tube with the triangular file and remove the tips.

3. Stand the tube (red-green end down) in the beaker; the red dot is pointed upward.

4. Allow the water sample to chromatograph up the tube.

5. A separation of a blue stain up the tube indicates the presence of cyanide. Other contaminants may cause the separation of a yellow, tan, or gray stain on the column.

Relative Measurement Tests

(i) pH

Apparatus: pH Meter

Reagent: Buffer, pH 7.0

Procedure:

1. Set up the pH Meter assembly and soak the electode in distilled water for at least 10 minutes before using. The liquid junction must always be below the water level.

2. Standardize the meter using Buffer, pH 7.0. Rinse the electrode thoroughly with distilled water.

3. Fill the beaker with the water sample and immerse the electrode.

4. Set the TEMP control to the approximate temperature of the sample.

5. Turn the POWER switch to the ON position. Record the pH value.

6. Turn the pH Meter off; rinse the electrode with distilled water and soak again.

(j) CONDUCTIVITY

Apparatus: Conductivity Meter with the Conductivity Meter Scale in place

Procedure:

1. Set the RANGE switch to position 5.

2. Plug the probe assembly into the 5 pin receptacle marked PROBE INPUT in the Spectrophotometer panel.

3. Immerse the probe in the water sample in the 50 ml beaker, or sampling container, sufficient to cover the vent holes.

4. Select the appropriate range beginning at the highest and working down. Each range on the meter scale corresponds to an equivalent range position. If the reading is in the lower 10 percent of the scale, switch to the next lower position.

5. Record the specific conductance in μ mhos/cm.

6. Return the RANGE switch to off.

(k) TURBIDITY

Apparatus: Spectrophotometer set at 450 nm with the Turbidity Meter Scale in place

Procedure:

1. Using a sample cell filled with a filtered water sample adjust the Spectrophotometer to read zero units using the LIGHT CONTROL.

2. Replace the filtered water sample with a second sample cell containing the unfiltered water sample. Record formazan turbidity units (FTU).

3. Turn the Spectrophotometer off.

(l) NITRATE NITROGEN

Apparatus: Spectrophotmeter set at 500 nm with the Nitrate Nitrogen Meter Scale in place

Reagents: NitraVer^{R} V Nitrate Powder Pillows

Procedure:

1. Use the graduate to add 25 ml of filtered water sample to a sample cell.

2. Add the contents of one NitraVer^{R} V pillow to the sample cell, stopper and shake vigorously for 1 minute. A positive test is indicated by an amber color. Allow 4-6 minutes for maximum color development.

3. Using a sample cell filled with filtered but untreated water sample adjust the LIGHT CONTROL to give a reading of zero units on the Spectrophotometer.

4. Replace with the cell containing the treated water sample. Record nitrate nitrogen in mg/ℓ.

5. Turn the Spectrophotometer off.

(m) COLOR

Apparatus: Spectrophotometer set at the "F" position with the Color Meter Scale in place

Procedure:

1. Using a sample cell filled with distilled water adjust the Spectrophotometer to read zero units using the LIGHT CONTROL.

2. Replace the distilled water with a sample cell containing the filtered water sample. Record units of color.

3. Turn the Spectrophotometer off.

(n) SULFATE

Apparatus: Spectrophotometer set at 450 nm with the Sulfate Meter Scale in place

Reagents: SulfaVer® IV Powder Pillows

Procedure:

1. Add 25 ml of filtered water sample to a sample cell using the graduate.

2. Add the contents of one SulfaVer® IV pillow to the cell and swirl to mix. A white turbidity indicates a positive test. Allow the turbidity to develop for 5-10 minutes.

3. Using a sample cell filled with distilled water adjust the Spectrophotometer to read zero units using the LIGHT CONTROL.

4. Replace with the cell containing the water sample. Record sulfate in mg/ℓ.

5. Turn the Spectrophotometer off.

(o) PHOSPHATE

Apparatus: Spectrophotometer set at 700 nm with the Phosphate Meter Scale in place

Reagents: PhosVer® III Powder Pillows

Procedure:

1. Add 25 ml of the filtered water sample to a sample cell using the graduate.

2. Add the contents of one PhosVer® III pillow and swirl to mix. A positive test is indicated by a blue-violet color. Allow 1-2 minutes for development.

3. Using a sample cell filled with distilled water adjust the Spectrophotometer to read zero units using the LIGHT CONTROL.

4. Replace with the cell containing the water sample. Record phosphate in mg/ℓ.

5. Turn the Spectrophotometer off.

(p) AMMONIA NITROGEN

Apparatus: Spectrophotometer set at 425 nm with the Ammonia Nitrogen Meter Scale in place

Reagents: Nesslers Reagent

Procedure:

1. Use the graduate to add 25 ml of distilled water to a sample cell.

2. Use the graduate to add 25 ml of filtered water sample to a second sample cell.

3. Add 1 ml of of Nesslers Reagent to each cell and swirl to mix. Ammonia nitrogen is indicated by a yellow color. Allow to stand for 5-10 minutes for maximum color development.

4. Using the sample cell containing distilled water adjust the Spectrophotometer to read zero units using the LIGHT CONTROL.

5. Replace with cell containing the water sample. Record ammonia nitrogen in mg/ℓ.

6. Turn the Spectrophotometer off.

Note: Some types of contaminants may cause a precipitate with this reaction. While it need not be measured it should be taken as a qualitative indicator of contamination.

(q) CHLORIDE

Apparatus: Spectrophotometer set at 463 nm with the Chloride Meter Scale in place

Reagent: Ferric Ion Solution
Mercuric Thiocyanate

Procedure:

1. Add 25 ml of distilled water to a sample cell using a graduate.

2. Add 25 ml of filtered water sample to a second cell using a graduate.

3. Add 1 ml of Ferric Ion Solution to each cell and swirl to mix.

4. Add 1 ml of Mercuric Thiocyanate to each cell and swirl to mix. A light orangish to reddish-brown color indicates a positive test. Allow 5-10 minutes for maximum development.

5. Using the distilled water sample adjust the Spectrophotometer to read zero units using the LIGHT CONTROL.

6. Replace the distilled water sample with a cell containing the water sample. Record chloride in mg/ℓ.

7. Turn the Spectrophotometer off.

(r) FLUORIDE

Apparatus: Spectrophotometer set at 580 nm with the Fluoride Meter Scale in place

Reagents: SPADNS Reagent

Procedure:

1. Add 25 ml of distilled water to a sample cell using the graduate.

2. Add 25 ml of filtered water sample to a second cell using the graduate.

3. Add 5 ml of SPADNS Reagent to each cell and swirl to mix. At high concentrations a bleaching of the red color may occur indicative of a positive test. At low concentrations this will not be perceptible visually but can be measured with the Spectrophotometer. Allow to stand one minute for maximum development.

4. Using the distilled water sample adjust the Spectrophotometer to read zero units using the LIGHT CONTROL.

5. Replace with the cell containing the water sample. Record fluoride in mg/ℓ.

6. Turn the Spectrophotometer off.

OUTLINE OF ANAYLTICAL PROCEDURES

Tests	Process	Comments/Interpretation
General Assessment a. Appearance	Describe water sample with respect to color, turbidity, suspended matter, slicks, etc.	
b. Color of Filtered Water	Filter 50 ml of water sample with the Filter Assembly. Note color.	Save reference sample in plastic tube. Save filter for next test.
c. Color of Suspended Matter	Note color of filter from previous test.	Save filter for reference.
Spot Tests d. Cholinesterase Inhibitors	20 ml water sample+1 ml Tris Buffer, pH 8.0 in 50 ml beaker. Wet round end of enzyme ticket with Tris Buffer. Stand square end in beaker for 5 min. Remove add substrate to each end.	After 2 min. round end (control) should be blue. Positive test if square end is white.
e. Benzene	Fill effervescent jar to level mark with water sample. Prepare tube (ends removed) in head assembly, arrow pointed out. Add Alka-Seltzer® tablet to jar, cap immediately.	Allow effervescence to finish. Positive test if white gel turns orange, tan, brown or greenish.
f. Heavy Metals	Add 2 ml Tris Buffer, pH 8.0 to plastic tube. Fill to level with water sample. Add 1 ml chloroform. Add 1 shot MercuVer™ 2. Shake 30-40 sec.	Positive test (chloroform layer) is pink, deep purple, yellow or orange. Negative test is blue-green or gray.
g. Phenol	Fill centrifuge tube to 25 ml mark with water sample. Add 1 ml Buffer Solution Hardness 1, 1 Phenol Reagent pillow and 1 Phenol 2 pillow. Add 1 ml chloroform. Shake 30-40 sec. Ignore ppt in water.	Positive test (chloroform layer) is orange to brown or rose to purple. Negative test is deep yellow.
h. Cyanide	Add 15 ml water sample to 50 ml beaker. Cyanide tube (ends removed) stood up in beaker, red dot up. Allow to chromatograph.	Blue stain if cyanide is present. Other contaminants yellow, tan or gray.
Relative Measurements i. pH	Soak electrode for 10 min. in distilled water. Set approximate temperature. Standardize with Buffer, pH 7.0. Measure and record pH of water sample. Rinse electrode, soak in distilled water.	

Tests	Process	Comments/Interpretation

Relative Measurements (Con't)

j. Conductivity — Plug probe into PROBE INPUT. Set RANGE to position 5. Immerse probe in water sample to cover vent holes. Switch to lower range until a reading is obtained. Record value.

Spectrophotometric Tests

Note: Tests requiring reagents should use water volumes measured with 25 ml graduate. Minimize on-time of spectrophotometer. Turn off after each reading.

	Wave Length	Blank Cell	Sample Cell	Process	Comments/Interpretation
k. Turbidity	450	Filtered Sample	Unfiltered Sample	Read directly, record value.	
l. Nitrate Nitrogen	500	Filtered Sample	Filtered Sample + Reagent	Add 1 NitraVer^{R}V pillow. Shake 1 min. Allow to stand 4-6 min. Record value.	Amber color indicates a positive test.
m. Color	F	Distilled Water	Filtered Sample	Read directly, record value.	
n. Sulfate	450	Distilled Water	Filtered Sample + Reagent	Add 1 SulfaVer^{D}IV pillow. Swirl to mix. Allow to stand 5-10 min. Record value.	White turbidity indicates a positive test. Clean cell with soap and water.
O. Phosphate	700	Distilled Water	Filtered Sample + Reagent	Add 1 PhosVer^{R}III pillow. Swirl to mix. Allow to stand 1-2 min. Record value.	Blue-violet color indicates positive test.
p. Ammonia Nitrogen	425	Distilled Water + Reagent	Filtered Sample + Reagent	Add 1 ml Nesslers Reagent. Swirl to mix. Allow to stand 5-10 min. Record value.	Yellow color indicates a positive test. Ppts may form with some contaminants.
q. Chloride	463	Distilled Water + Reagents	Filtered Sample + Reagents	Add 1 ml Ferric Ion Solution. Add 1 ml Mercuric Thiocyanate. Swirl to mix. Allow to stand 5-10 min. Record value.	Lt. orange to reddish brown color indicates a positive test.
r. Fluoride	580	Distilled Water + Reagent	Filtered Sample + Reagent	Add 5 ml SPADNS. Swirl to mix. Allow to stand 1 min. Record value.	Bleaching may occur at high high conc. not perceptible at low conc.

SECTION V
CONSUMABLE MATERIALS

Cat. No.	Description	Unit	Unit Price	Source
2387-20	Mercu Ver™2	1 oz.	$5.25	Hach Chemical Co. P.O. Box 907, Ames, IA 50010
151-13	Nesslers Reagent	4 oz.	2.85	Hach Chemical Co.
2125-99	Phos Ver® III Powder Pillows	pkg of 100	8.00	Hach Chemical Co.
14034-99	Nitra Ver® V Powder Pillows	pkg of 100	6.95	Hach Chemical Co.
12065-99	Sulfa Ver® IV Powder Pillows	pkg of 100	6.50	Hach Chemical Co.
1836-99	Phenol 2 Reagent Powder Pillows	pkg of 100	6.00	Hach Chemical Co.
424-16	Buffer Solution, Hardness 1	qt	4.00	Hach Chemical Co.
444-16	SPADNS Reagent	qt	6.60	Hach Chemical Co.
872-99	Phenol Reagent Powder Pillows	pkg of 100	15.00	Hach Chemical Co.
2062-32	Reference Electrode Filling Solution	2 oz.	2.35	Hach Chemical Co.
Gastec No. 121	Benzene Detector Tube	pkg of 10	7.20	National Environmental Instruments, Inc. P.O. Box 590, Warwick, RI 02888
8104-204	Detector Tube, Kitagawa Cyanide Ion	pkg of 10	15.00	Matheson Gas Products P.O. Box 85, East Rutherford, NJ 07073

SECTION V
CONSUMABLE MATERIALS (continued)

Cat. No.	Description	Unit	Unit Price	Source
61693	Prefilters, Type A Glass Fiber, 25 mm	pkg of 500	$15.00	Gelman Instrument Co. 600 Sourth Wagner Road Ann Arbor, MI 48106
	Shell Container, Clear cellulose acetate, 3/8 in. diam x 3-½ in. length	1000	15.72	Flex Products Corp. 445 Industrial Road Carlstadt, NJ 07072
	Printing for above, single line 1/32 in. wide around circumference 1-3/16 in. from top (open end) and the words "WATER LEVEL" in paraliel slightly above the line.	1000	10.30	Flex Products Corp.
	Plug, natural polyethylene, for 3/8 in. diameter shell container	1000	3.62	Flex Products Corp.
FSN 6665-00-909-3647	Refill Kit, Chemical Agent Detector, ABC-M30A1	each	20.57	Headquarters, ARMCOM AMSAR-MMD-L Rock Island, IL 61201
	Buffer, pH 7.0			Chemical Supply House
	Chloroform			Chemical Supply House
	Dye Marker			Chemical Supply House
	Liquid Soap (Phosphorous - free)			Chemical Supply House

SECTION V
CONSUMABLE MATERIALS (continued)

Cat. No.	Description	Unit	Unit Price	Source
	Alka-Seltzer^{R} (Without Aspirin)	pkg of 20	$ .60	Drugstore
	Battery, 6.75 V, Mallory TR-235R	each	5.85	Electronic Supply House
	Battery, 1.5 V, D Size, Alkaline	each	1.00	Electronic Supply House
	Battery, 1.5 V, AA Size, Alkaline	each	.75	Electronic Supply House
	Distilled Water			Laboratory
	Tris Buffer, pH 8.0			Laboratory
	Ferric Ion Solution			Laboratory
	Mercuric Thiocyanate Solution			Laboratory

REFERENCES

Silvestri, A., Goodman, A., McCormack, L. M., Razulis, M. K., Jones, A. R., Jr. and Davis, M. E. P. Development of a Kit for Detecting Hazardous Material Spills in Waterways. Interagency Agreement No. EPA-IAG-0546. Chemical Systems Laboratory, ARRADCOM, Aberdeen Proving Ground, MD 21010.

Hach Direct Reading Engineer's Laboratory, Model DR-EL/2, Methods Manual.

Laboratory pH Meter, Model 1975 and 2075.